HOMOGENOUS AND CARBON GRADED HIGH MANGANESE STEELS

HOMOGENOUS AND CARBON GRADED HIGH MANGANESE STEELS

By

Morteza Ghasri Khouzani, B.Sc., M.Sc.,

A Thesis

Submitted to the School of Graduate Studies in Partial Fulfilment of the Requirements for the Degree Doctor of Philosophy

McMaster University

© Copyright by Morteza Ghasri Khouzani, March 2015

Doctor of Philosophy (2015)

McMaster University

(Materials Science & Engineering)

Hamilton, Ontario

TITLE:	Homogenous and Carbon graded High Manganese Steels
AUTHOR:	Morteza Ghasri Khouzani, B.Sc., M.Sc., (Isfahan University of
	Technology)
SUPERVISOR:	Dr. Joseph R. McDermid
NUMBER OF PAGES:	xxii, 227

ABSTRACT

High manganese steels offer an outstanding combination of high strength and ductility owing to their high sustained strain hardening rates. The strain-induced deformation products in these steels (mechanical twins and ε -martensite) increase the work hardening rates by acting as barriers for dislocation motion. A significant determinant of the deformation products in these steels is the value of stacking fault energy (SFE), which is in turn strongly dependent on the alloy manganese and carbon contents. The main objective of present work is to investigate the microstructural evolution and mechanical behaviour of both homogenous and compositionally graded high-Mn steels, where C compositional gradients were introduced into the latter.

The starting materials in this work were cold rolled Fe-22Mn-0.6C and Fe-30Mn-0.6C steels. For both starting alloys, decarburization and annealing heat-treatments were used to obtain four homogenous alloys with carbon contents of approximately 0, 0.2, 0.4 and 0.6 wt. % with similar grain sizes. Decarburization heat treatments were also applied to obtain three carbon graded Fe-22Mn-C alloys (G1, G2, G3) and one carbon graded Fe-30Mn-C alloy. Microstructural observations determined the deformation products to be mechanical ε-martensite for the 22Mn-0C and 22Mn-0.2C alloys and mechanical twins for the 22Mn-0.6C, 30Mn-0.2C, 30Mn-0.4C and 30Mn-0.6C alloys. For the 22Mn-0.4C and 30Mn-0C alloys, both mechanical twins and ε-martensite were observed during deformation. For all the carbon graded Fe-22Mn-C alloys, the dominant deformation products changed from mechanical ε-martensite at the near-surface layer to a mixture of mechanical twins and ε-martensite or mechanical twins only at the inner cross-section layers. In the case of carbon graded Fe-30Mn-C alloy, the deformation products altered from a combination of mechanical ε -martensite and twins at the near-surface layer to mechanical twins at the inner cross-section layers.

For all the homogenous alloys, the ultimate tensile strength and uniform elongation increased with increasing alloy carbon content. The work hardening behaviour of these steels was successfully modelled using a modified Kocks-Mecking model, in which the work hardening was the sum of the dislocation glide contribution and the phase transition contribution – mechanical twinning and/or mechanical ε-martensite formation – as dictated by the formation kinetics of both deformation products. For both alloy systems, the mechanical properties of the carbon graded alloys were not as good as the monolithic 22Mn-0.6C and 30Mn-0.6C alloys due to their lower sustained high work hardening rates.

Both the mechanical ε -martensite and twin formation were found to follow a sigmoidal kinetic with strain. In the case of twin formation homogenous alloys, the saturated volume fraction of twins was directly proportional to the alloy SFE. For the ε -martensite formation homogenous alloys, the ε -martensite volume fraction at fracture was found to be strongly dependent on alloy SFE, where it declined sigmoidally with increasing alloy SFE. It was also found that the ε -martensite volume fraction at fracture – approximately 0.7 – was independent of SFE for SFE $\leq 6 \text{ mJ/m}^2$. This indicated that the critical damage mechanism was determined by the kinetics of the ε -martensite formation, which was in turn dictated by the alloy SFE. Finally, it was found that the stress for the

onset of mechanical twinning – and consequent increase in the work hardening rate – for the higher SFE, twinning dominated alloys was linearly proportional to the alloy SFE.

ACKNOWLEDGEMENTS

Most importantly I am extremely grateful to my supervisor, Dr. Joseph McDermid, for providing me with tireless and excellent guidance. I acknowledge his generosity in letting me perform many expensive experiments and analyses, which made me the most expensive student (the title was previously held by Dr. Erika Bellhouse). I appreciate his continuous support and encouragement throughout my Ph.D. I would also like to thank my supervisory committee members, Dr. Gianluigi Botton and Dr. Hatem Zurob, for their insightful comments and discussions. I am truly indebted to Dr. Hatem Zurob for his generosity in allowing me to use his facilities.

I would like to acknowledge the following people for their assistance with the experimental and analytical work: Dr. Connie Barry and Dr. Mike Bruhis from the Centre for Automotive Materials and Corrosion (CAMC), Chris Butcher and Andy Duft from the Canadian Centre for Electron Microscopy (CCEM), Dr. James Britten and Victoria Jarvis from the McMaster Analytical X-Ray Facility (MAX), Doug Culley, Xiaogang Li and Ed McCaffery. I would also like to thank the Resource for the Innovation of Engineered Materials (RIEM) program of CANMETMaterials for fabrication of the Fe-22Mn-0.6C and Fe-30Mn-0.6C sheet steels used in this study.

I would like to thank the Natural Sciences and Engineering Research Council of Canada (NSERC) and the members of the McMaster Steel Research Centre for their financial support.

Lastly, I am thankful to my family and friends for their support.

TABLE OF CONTENTS

ABS	STRACI	`iii
ACI	KNOWL	EDGEMENTSvi
TAI	BLE OF	CONTENTSvii
LIS'	T OF FI	GURESxii
LIS'	T OF TA	BLESxxii
1	INTRO	DUCTION1
2	LITER	ATURE REVIEW4
2.1	Sta	cking Fault Energy (SFE)4
	2.1.1	Calculations of SFE
	2.1.2	Factors Affecting SFE10
	2.1.3	SFE Dependence of Deformation Products
2.2	Tw	inning17
2.3	Ma	rtensitic Transformation19
2.4	Stra	ain Hardening25
2.5	Hig	h Manganese TWIP Steels
	2.5.1	Dynamic Hall-Petch Effect
	2.5.2	Dynamic Strain Aging (DSA) Effect
	2.5.3	Work Hardening Behaviour

Ph.l	D. Thesis	– M. Ghasri Khouzani McMaster – Materials Science & Engineering.	
	2.5.4	Effect of Chemical Composition on Tensile Properties	
	2.5.5	Role of Grain Size	
2.6	Hig	h Manganese TRIP Steels	
	2.6.1	Mechanical Behaviour	
	2.6.2	Microstructural Evolution	
	2.6.3	Fracture Mechanism	
2.7	Hig	h Manganese TWIP/TRIP Steels49	
3	OBJEC	TIVES	
4	EXPER	RIMENTAL METHODS52	
4.1	Star	ting Materials	
4.2	.2 Heat Treatments		
4.3	Sam	ple Preparation55	
4.4	Med	chanical Testing	
4.5	Cha	racterization Techniques58	
	4.5.1	Glow Discharge Optical Emission Spectroscopy (GDOES)	
	4.5.2	Inductively Coupled Plasma - Optical Emission Spectroscopy (ICP -	
	OES)		
	4.5.3	Carbon and Sulfur Combustion Analysis60	

Ph.I	D. Thesis	s – M. Ghasri Khouzani McMaster – Materials Science & Engineering.
	4.5.4	Scanning Electron Microscopy (SEM) and Electron Backscatter Diffraction
	(EBSD)60
	4.5.5	X-Ray Diffraction (XRD)62
4.6	Fra	cture Analysis62
5	EXPE	RIMENTAL RESULTS FOR FE-22MN-C ALLOYS63
5.1	Но	mogenous Fe-22Mn-C Alloys63
	5.1.1	As Heat-Treated Alloy Carbon Content and Microstructure
	5.1.2	Tensile Behaviour
	5.1.3	Microstructural Evolution with Tensile Deformation71
	5.1.4	Fractography
	5.1.5	Summary of Homogenous Fe-22Mn-C Alloys
5.2	Car	rbon Graded Fe-22Mn-C Alloys90
	5.2.1	As Decarburized Alloy Carbon Concentration Profiles and SFE90
	5.2.2	As Heat-Treated Microstructures94
	5.2.3	Tensile Behaviour96
	5.2.4	Microstructural Evolution with Tensile Deformation100
	5.2.5	Fractography113
	5.2.6	Summary of Carbon Graded Fe-22Mn-C Alloys121
6	EXPE	RIMENTAL RESAULTS FOR FE-30MN-C ALLOYS123

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.		
6.1	Ho	mogenous Fe-30Mn-C Alloys
	6.1.1	As Heat-Treated Alloy Carbon Content and Microstructure
	6.1.2	Tensile Behaviour
	6.1.3	Microstructural Evolution with Tensile Deformation132
	6.1.4	Fractography143
	6.1.5	Summary of Homogenous Fe-30Mn-C Alloys148
6.2	Car	bon Graded Fe-30Mn-C Alloy149
	6.2.1	As Decarburized Alloy Carbon Concentration Profile and SFE149
	6.2.2	As Heat-Treated Microstructure
	6.2.3	Tensile Behaviour
	6.2.4	Microstructural Evolution with Tensile Deformation158
	6.2.5	Fractography164
	6.2.6	Summary of Carbon Graded Fe-30Mn-C Alloy167
7	DISCUSSION169	
7.1	Cor	nparison of Deformation Product Formation between Homogenous Fe-
22N	In-C and	Fe-30Mn-C Alloys
	7.1.1	Mechanical Twinning169
	7.1.2	Mechanical Martensitic Transformation174
	7.1.3	Combination of Mechanical Twinning and Martensitic Transformation 177

Ph.1	D. Thesis	s – M. Ghasri Khouzani McMaster – Materials Science & Engineering	g.
7.2	Mo	odelling the Work Hardening Rate of Homogenous Alloys17	79
	7.2.1	ε-Martensite Formation Alloys18	31
	7.2.2	Twin Formation Alloys18	35
	7.2.3	Mixed Deformation Product Alloys19) 1
7.3	Co	omparison between the Mechanical Properties of the Homogenous and Carbo	on
Gra	ded Allo	Dys19) 7
8	CONC	CLUSIONS)0
8.1	Но	omogenous Fe-22Mn-C Alloys20)0
8.2	Ca	urbon Graded Fe-22Mn-C Alloys20)1
8.3	Но	omogenous Fe-30Mn-C Alloys20)2
8.4	Car	rbon Graded Fe-30Mn-C Alloy20)2
RE	FEREN	CES)4
App	oendix A	A: Repeatability of Tensile Results for the Experimental Alloys	18
App	oendix B	3: Microstructural Evolution Results for the Carbon Graded G1 Alloy22	23
App	oendix C	22 Fractography Results for the Carbon Graded G1 Alloy	26

LIST OF FIGURES

Figure 1-1: Ductility and strength of several classes of steels (Bleck and Phiu-On, 2005).1 Figure 2-1: The stacking fault is assumed to be an interface separating two rigidly displaced matrix areas; (a) infinite stacking fault illustrated by stacking operators; (b) terminated stacking fault bounded by two partial dislocations (Ferreira and Müllner, Figure 2-2: The stacking fault is considered as a separate phase consisting of two layers with a different stacking sequence; (a) infinite stacking fault; (b) terminated stacking fault (Ferreira and Müllner, 1998)......7 Figure 2-3: Variation of SFE as a function of Mn content in Fe-Mn alloys (Lee and Choi, Figure 2-4: Calculated SFE as a function of temperature for Fe-22Mn-0.6C steel (Allain Figure 2-5: The SFE values as a function of temperature for Fe-22Mn-0.6C alloy; data was derived using Allain et al. and Dumay et al. SFE models (Allain et al., 2004b, Dumay Figure 2-7: Schematic of the different forms of martensitic transformations (Olson and Figure 2-8: Transformation temperatures as a function of carbon concentration in Fe-

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.
Figure 2-9: Schematic illustration of nucleation mechanism for ε martensite (Bracke et
al., 2006)
Figure 2-10: High resolution image showing the layered substructure of a large ε -
martensite plate (Putaux and Chevalier, 1996)25
Figure 2-11: $(\sigma_V/\mu)^{1/2}$ versus $\left[\frac{kT}{\mu b^3}Ln(\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}})\right]^{1/2}$ plots for some FCC metals. χ is the stacking
fault energy (SFE) value (Kocks and Mecking, 2003)
Figure 2-12: Schematic illustration of dynamic Hall-Petch effect (De Cooman et al.,
2009)
Figure 2-13: Serrations in tensile curve of Fe-20Mn-1.2C TWIP steel tested at room
temperature (Renard et al., 2010)
Figure 2-14: Dastur-Leslie model for DSA in Hadfield steels (De Cooman et al., 2009).32
Figure 2-15: Work hardening rate as a function of true strain for Fe-20Mn-1.2C TWIP
steel tensile tested at room temperature; G is the shear modulus (Renard and Jacques,
2012)
Figure 2-16: Twin volume fraction as a function of true strain for Fe-20Mn-1.2C TWIP
steel (Renard and Jacques, 2012)
Figure 2-17: True stress-true strain curves of several high manganese TWIP steels as well
as Fe-30 Mn alloy (as reference material) (Bouaziz et al., 2011b)37
Figure 2-18: Engineering stress-engineering strain plots of Fe-22Mn-0.6C TWIP steel as
a function of grain size (Bouaziz et al., 2011a)
Figure 2-19: Grain size effect on strain and stress of twinning onset (Bouaziz et al., 2008).

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering
Figure 2-20: True stress increment versus true strain for Fe-20Mn-3Si-3Al TRIP stee
(Grässel et al., 2000)
Figure 2-21: Tensile behaviour of Fe-24Mn-0C alloy at room temperature (Liang, 2008)
Figure 2-22: True stress-true strain curves and corresponding work hardening plots of the
Fe-17Mn-0.6C steel at 173 K and 223 K (Koyama et al., 2011)42
Figure 2-23: Volume fraction evolution of present phases with true strain for Fe-20Mn
3Si-3Al TRIP steel (Grässel et al., 2000)
Figure 2-24: Evolution of ɛ-martensite volume fraction with tensile strain for Fe-24Mn
0C TRIP steel (Liang, 2008)
Figure 2-25: ɛ-martensite volume at fracture as a function of strain rate for Fe-12Mn-0.60
steel (Lee et al., 2014)
Figure 2-26: Fracture surface of Fe-17Mn-0.6C alloy after tensile testing at 173 K (a
(Koyama et al., 2011) and schematic illustration of step-like ridge formation (b) (Takak
et al., 1990)
Figure 2-27: SEM fractographs of Fe-24Mn-0C alloy after uniaxial tensile test at 293 k
(Liang, 2008)
Figure 3-1: Schematic illustration of carbon gradient within thickness of carbon graded
steels
Figure 4-1: SEM micrographs from the microstructure of as-received steels
Figure 4-2: Sample geometry for all tensile tests (all dimensions are in mm and the
sample thickness is the same as the sheet thickness)

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.
Figure 5-1: Typical EBSD phase maps from the cross-section of as heat-treated
homogenous Fe-22Mn-C alloys
Figure 5-2: Room temperature true stress-true strain curves for the homogenous Fe-
22Mn-0C alloy
Figure 5-3: Room temperature tensile response of homogenous Fe-22Mn-C alloys69
Figure 5-4: Evolution of ϵ -martensite volume fraction with tensile strain for all
homogenous Fe-22Mn-C alloys, measured via XRD72
Figure 5-5: EBSD phase maps of cross-sections for 0C alloy as a function of true strain.
Figure 5-6: EBSD phase maps of cross-sections for 0.2C alloy as a function of true strain.
Figure 5-7: EBSD phase maps of cross-sections for 0.4C alloy as a function of true strain.
Figure 5-8: Twin volume fraction as a function of strain for the 0.4C and 0.6C alloys80
Figure 5-9: EBSD phase maps of cross-sections for 0.6C alloy as a function of true strain.
Figure 5-10: SEM fractographs of homogenous Fe-22Mn-0C alloy
Figure 5-11: SEM fractographs of homogenous Fe-22Mn-0.2C alloy
Figure 5-12: SEM fractographs of homogenous Fe-22Mn-0.4C alloy87
Figure 5-13: SEM fractographs of homogenous Fe-22Mn-0.6C alloy

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.
Figure 5-14: Carbon concentration profile for the carbon graded Fe-22Mn-C steels. Note
that the total sheet thickness was 1.5 mm or 1500 microns and that symmetry was
assumed91
Figure 5-15: Manganese concentration profile for the carbon graded Fe-22Mn-C alloys.92
Figure 5-16: SFE versus sample depth for all carbon graded Fe-22Mn-C steels based on
the Dumay et al. SFE model (Dumay et al., 2008)93
Figure 5-17: EBSD phase maps from cross-section of as decarburized carbon graded Fe-
22Mn-C steels95
Figure 5-18: Tensile response of carbon graded Fe-22Mn-C alloys at room temperature.
Figure 5-19: EBSD phase maps from the near surface cross-section (0-250 microns from
the surface) of the G2 alloy as a function of true strain101
Figure 5-20: Evolution of ε -martensite volume fraction with applied strain for G2 and G3
alloys; data was obtained from EBSD phase maps using the HKL software102
Figure 5-21: EBSD phase maps from the mid-thickness cross-section (250-500 microns
from the surface) of the G2 alloy as a function of true strain104
Figure 5-22: Twin volume fraction as a function of strain for the G2 and G3 alloys105
Figure 5-23: EBSD phase maps from the core cross-section (500-750 microns from the
surface) of G2 alloy as a function of true strain107
Figure 5-24: EBSD phase maps from the near surface cross-section (0-250 microns from
the surface) of G3 alloy as a function of true strain109

Ph.D. Thesis – M. Ghasri Khouzani	McMaster – Materials Science & Engineering.
Figure 5-25: EBSD phase maps from the	e mid-thickness cross-section (250-500 microns
from the surface) of G3 alloy as a function	n of true strain111
Figure 5-26: EBSD phase maps from the	e core cross-section (500-750 microns from the
surface) of G3 alloy as a function of true s	train113
Figure 5-27: SEM micrographs from fract	ure surface of G2 alloy115
Figure 5-28: SEM micrographs from cros	ss-section near fracture surface, sectioned along
tensile direction, of G2 alloy	
Figure 5-29: SEM micrographs from fract	ure surface of G3 alloy119
Figure 5-30: SEM micrographs from cross	ss-section near fracture surface, sectioned along
tensile direction, of G3 alloy	
Figure 6-1: Typical EBSD phase map	os from the cross-section of as heat-treated
homogenous Fe-30Mn-C alloys	
Figure 6-2: Room temperature true stre	ss-true strain curves for the homogenous Fe-
30Mn-0C alloy	
Figure 6-3: Room temperature tensile resp	oonse of homogenous Fe-30Mn-C alloys 129
Figure 6-4: Evolution of <i>ɛ</i> -martensite	volume fraction with tensile strain for the
homogenous Fe-30Mn-0C alloy, measured	d via XRD133
Figure 6-5: EBSD phase maps of cross-	sections of the 0C alloy as a function of true
strain	
Figure 6-6: Twin volume fraction as a	function of strain for all the homogenous Fe-
30Mn-C alloys.	

Ph.D. Thesis – M. Ghasri Khouzani N	IcMaster – Materials Science & Engineering.
Figure 6-7: EBSD phase maps of cross-section	ons for 0.2C alloy as a function of true strain.
Figure 6-8: EBSD phase maps of cross-section	ons for 0.4C alloy as a function of true strain.
Figure 6-9: EBSD phase maps of cross-section	ons for 0.6C alloy as a function of true strain.
Figure 6-10: SEM fractographs of homogene	ous Fe-30Mn-0C alloy144
Figure 6-11: SEM fractographs of homogene	ous Fe-30Mn-0.2C alloy145
Figure 6-12: SEM fractographs of homogene	ous Fe-30Mn-0.4C alloy146
Figure 6-13: SEM fractographs of homogene	ous Fe-30Mn-0.6C alloy147
Figure 6-14: Carbon concentration profile for	or the carbon graded Fe-30Mn-C steel. Note
that the total sheet thickness was 1.6 mm	or 1600 microns and that symmetry was
assumed	
Figure 6-15: Manganese concentration prof	ile for the carbon graded Fe-30Mn-C alloy.
Figure 6-16: SFE versus sample depth for c	arbon graded Fe-30Mn-C steel based on the
Dumay et al. (Dumay et al., 2008) SFE mode	el152
Figure 6-17: EBSD phase maps from cross-	section of as decarburized carbon graded Fe-
30Mn-C steel	
Figure 6-18: Tensile response of carbon grad	ed and monolithic alloys at RT156
Figure 6-19: EBSD phase maps from the ne	ar surface cross-section (0-250 microns from
the surface) of carbon graded alloy as a funct	ion of true strain159

Ph.D. Thesis – M. Ghasri Khouzani	McMaster – Materials Science & Engineering.
Figure 6-20: Twin volume fraction as a fu	nction of strain for the carbon graded alloy160
Figure 6-21: EBSD phase maps from the	e mid-thickness cross-section (250-500 microns
from the surface) of carbon graded alloy a	s a function of true strain162
Figure 6-22: EBSD phase maps from the	e core cross-section (500-750 microns from the
surface) of carbon graded alloy as a functi	on of true strain164
Figure 6-23: SEM micrographs from the	fracture surface of the carbon graded Fe-30Mn-
C alloy	
Figure 6-24: SEM micrographs from cros	ss-section near fracture surface, sectioned along
tensile direction, of carbon graded Fe-30M	In-C alloy167
Figure 7-1: Stress for the initiation of	twinning as a function of SFE following the
proposed model for of Byun (Byun, 20	03), supplemented by the data of Renard and
Jacques (Renard and Jacques, 2012), Yan	ng (Yang, 2010) and Jin and Lee (Jin and Lee,
2009)	
Figure 7-2: Saturated twin volume fractio	n as a function of SFE for various TWIP alloys.
Figure 7-3: Saturated ε-martensite volume	e fraction as a function of SFE for various high-
Mn Fe-Mn-C alloys	
Figure 7-4: Sum of ε-martensite and twin	volume fractions versus true strain for the Fe-
22Mn-0.4C, Fe-22Mn-0.6C, Fe-30Mn-0.4	C and Fe-30Mn-0.6C alloys, including volume
fraction of twins at fracture from Renard	and Jacques (Renard and Jacques, 2012) and Lu
et al. (Lu et al., 2014)	

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.
Figure 7-5: Fitting plots of ε -martensite volume fraction versus true stress for the Fe-
22Mn-0C and Fe-22Mn-0.2C alloys
Figure 7-6: Comparison between the work hardening experimental results and model (Eq.
7-4) for the Fe-22Mn-0C and Fe-22Mn-0.2C alloys
Figure 7-7: Fitted plots of twin volume fraction evolution with true stress for the twin
formation alloys; symbol lines denote experimental results and solid lines are fitted
curves
Figure 7-8: Comparison between the work hardening experimental results and model (Eq.
7-4) for the Fe-22Mn-0.6C and Fe-30Mn-0.2C alloys
Figure 7-9: Comparison between the work hardening experimental results and model (Eq.
7-4) for the Fe-30Mn-0.4C and Fe-30Mn-0.6C alloys
Figure 7-10: Fitted plots of the deformation product evolution with true stress in the Fe-
22Mn-0.4C and Fe-30Mn-0C alloys
Figure 7-11: Comparison between the work hardening experimental results and model
(Eq. 7-4) for the Fe-22Mn-0.4C and Fe-30Mn-0C alloys
Figure 7-12: Schematic illustration of the carbon graded alloys as laminar composite.
Assuming symmetry, only the surface to the core is illustrated
Figure A-1: Room temperature true stress-true strain curves for the homogenous Fe-
22Mn-0.2C and Fe-22Mn-0.4C alloys
Figure A-2: Room temperature true stress-true strain curves for the homogenous Fe-
22Mn-0.6C and carbon graded G1 alloys

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.
Figure A-3: Room temperature true stress-true strain curves for the carbon graded G2 and
G3 alloys
Figure A-4: Room temperature true stress-true strain curves for the homogenous Fe-
30Mn-0.2C and Fe-30Mn-0.4C alloys
Figure A-5: Room temperature true stress-true strain curves for the homogenous Fe-
30Mn-0.6C and carbon graded Fe-30Mn-C alloys222
Figure B-1: EBSD phase maps from the near surface cross-section (0-250 microns from
the surface) of G1 alloy as a function of true strain
Figure B-2: EBSD phase maps from the mid-thickness cross-section (250-500 microns
from the surface) of G1 alloy as a function of true strain
Figure B-3: EBSD phase maps from the core cross-section (500-750 microns from the
surface) of G1 alloy as a function of true strain
Figure C-1: SEM micrographs from fracture surface of the carbon graded G1 alloy226
Figure C-2: SEM micrographs from cross-section near fracture surface, sectioned along
tensile direction, of the carbon graded G1 alloy227

LIST OF TABLES

Table 4-1. Chemical composition of the as-received steels
Table 4-2. Heat treatments parameters for Fe-22Mn-C alloys. 54
Table 4-3. Heat treatments parameters for Fe-30Mn-C alloys. 55
Table 5-1. Carbon content and computed SFE values for all homogenous Fe-21.8Mn-C
alloys64
Table 5-2. Room temperature tensile characteristics of homogenous Fe-22Mn-C alloys. 70
Table 5-3. Decarburization conditions for carbon graded Fe-22Mn-C alloys
Table 5-4. Room temperature tensile characteristics of the carbon graded Fe-22Mn-C
alloys
Table 6-1. Carbon content and computed SFE values for all homogenous Fe-30Mn-C
alloys124
Table 6-2. Room temperature tensile characteristics of homogenous Fe-30Mn-C alloys.
Table 6-3. Room temperature tensile characteristics of the carbon graded Fe-30Mn-C and
monolithic Fe-30Mn-0.6C alloys157
Table 7-1. Numerical values of parameters used for modelling the work hardening of all
homogenous Fe-22Mn-C alloys196
Table 7-2. Numerical values of parameters used for modelling the work hardening rate as
a function of various homogenous Fe-22Mn-C alloys
Table 7-3. Numerical values for parameters used for modelling the work hardening rate as
a function of various homogenous Fe-30Mn-C alloys

1 INTRODUCTION

The development of ultra high strength steels with high ductility and toughness has long been pursued by the steel industry. As a result, especially for automotive sheet steels, several advanced steels have been introduced such as Dual Phase (DP), low alloy TRansformation-Induced Plasticity (TRIP) steels and high manganese steels. Figure 1-1 shows an overview of the ultimate tensile strength (UTS) and total elongation of a wide range of commonly used steels. It is clearly seen that compared to other steels, high-Mn steels have a superior combination of both a high UTS and a large total elongation.



Figure 1-1: Ductility and strength of several classes of steels (Bleck and Phiu-On, 2005).

An early high manganese steel, containing approximately 1.2 wt. % C and 13 wt. % Mn, was invented by Sir Robert Hadfield in 1882 (Tweedale, 1985). This alloy, now

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

commonly known as Hadfield steel, rapidly gained acceptance as a useful engineering material primarily in the field of high wear applications such as earthmoving, mining, quarrying and oil well drilling. Unfortunately, for sheet applications the high (>1 wt. %) carbon levels in Hadfield steels meant that they were difficult to process (due to carbide precipitation) and had very limited weldability (Mendez et al., 2004). To address these issues, more modern alloys have reduced their carbon concentrations to 0-0.6 wt. % and compensated for this C reduction effect on austenite stabilization with an increase in the manganese level to 15-33 wt. %. Also, modern high Mn steels commonly contain relatively small amounts of other alloying elements such as aluminum and silicon to aid in property development. This shift in alloy compositions away from that of the original Hadfield steel has lead to a new class of modern high manganese steels (Bouaziz and Guelton, 2001, Bouaziz et al., 2011, Grässel et al., 2000, Kim et al., 2011, Lee, 2012, Liang et al., 2009, Sahu et al., 2012).

The property requirements for selected automobile applications are (Hulka, 2004):

- high strength: allows the automakers to reduce the vehicle weight – through reduced sheet thickness,

- high formability: forming of complex geometries with fewer deformation steps,

- high energy absorption: increased safety when used in crash relevant components,

- cost reduction: replacement of cost intensive materials (Al, Mg, stainless steel) or cost-intensive processes (hot pressing).

In the case of high-Mn steels, these requirements are met due to their sustained high work hardening rates during the majority of their plastic deformation. The high rates of work Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

hardening in these steels have been attributed to strain-induced deformation products such as mechanical twins and ε -martensite. These are the so called twinning induced plasticity (TWIP) and transformation induced plasticity (TRIP) effects, respectively (Bracke et al., 2006, Frommeyer et al., 2003, Grässel et al., 2000, Remy, 1978, Remy and Pineau, 1977). The mechanical twin and ε -martensite formation also results in an effective grain size refinement and reduction in dislocation mean free path often referred to as the "dynamic Hall-Petch" effect (Allain et al., 2004a, Allain et al., 2004c, Bouaziz et al., 2008, Bouaziz et al., 2011a, Bouaziz and Guelton, 2001, Gutierrez-Urrutia and Raabe, 2012).

The main objective of present work is to investigate the microstructural evolution and mechanical behaviour of both homogenous and compositionally graded high-Mn steels, where C compositional gradients were introduced into the latter. To achieve this goal, two high-Mn alloys were chosen: Fe-22Mn-0.6C and Fe-30Mn-0.6C. The present thesis comprises seven chapters. In Chapter 2, a critical literature review on the fundamental aspects of the present work will be presented. The main objectives of the present work will be presented in Chapter 3. In Chapter 4, the experimental methods and techniques that were applied in this work will be described. The experimental results for the homogenous and C-graded Fe-22Mn-C and Fe-30Mn-C alloy systems will be presented in Chapter 5 and Chapter 6, respectively. In Chapter 7, the combined experimental results for both alloys will be discussed in detail. The conclusions of the present work will be presented in Chapter 8.

2 LITERATURE REVIEW

In this chapter, the fundamental aspects of high-manganese steels will be reviewed by starting off with stacking fault energy (SFE), which has a major influence on the deformation products in high-Mn steels. Then, the ideas on the phase transitions in the high-Mn alloys – i.e. twinning and martensitic transformation – will be described followed by a review of isotropic and kinematic strain hardening. Finally, relatively recent papers on microstructure, mechanical behaviour and fracture mechanisms of high-Mn alloys with various deformation products will be reviewed.

2.1 Stacking Fault Energy (SFE)

As fabricated, modern high-Mn steels are fully austenitic, meaning that at equilibrium they have a face-centered cubic (FCC) microstructure. However, modern high Mn steels, depending on their composition, have a variety of stacking fault energies (SFE) which can result in a variety of deformation products – in this case twins and HCP ϵ martensite – appearing during deformation processes. It is, in part, the type and population of these deformation products which determines the microstructural and mechanical property evolution of these alloys. For this reason, it is necessary to understand the fundamentals of stacking faults and stacking fault energies to contextualize the observations and literature of high-Mn steels.

A stacking fault is a planar defect and, as its name implies, it is a local region in which the regular crystallographic stacking sequence has been interrupted or changed. The ideal stacking sequence of the close-packed {111} planes in the FCC structure, Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

ABCABC..., can be disturbed in various ways. If an additional plane is inserted into the stacking sequence, the resultant defect is called an extrinsic stacking fault, i.e. ABCABACABC. On the other hand, if a plane is removed from the stacking sequence, i.e. the stacking order is ABCACABC, an intrinsic stacking fault is present. This fault occurs when the perfect a/2 <110> dislocation dissociates into two a/6 <211> dislocations, known as Shockley partial dislocations (Kelly et al., 2000):

$$\frac{a}{2}[01\bar{1}] \to \frac{a}{6}[11\bar{2}] + \frac{a}{6}[\bar{1}2\bar{1}]$$
(2-1)

Since the dislocation energy is proportional to the square of the magnitude of the Burgers vector $(\left|\vec{b}\right|^2)$, this dissociation is energetically favoured (Kelly et al., 2000). The two partial dislocations repel each other with a force due to their elastic interaction. On the other hand, the stacking fault energy (SFE) provides a force Γ per unit length of the line tending to pull the partial dislocations together. In this case, an equilibrium separation d will be established when the repulsive and attractive forces balance. The approximate equilibrium separation can be calculated using (Schoeck, 1999):

$$d = \frac{\mu b^2}{4\pi\Gamma} \tag{2-2}$$

where μ is shear modulus. The equilibrium separation *d* of two partials is inversely proportional to the stacking fault energy. Thus, the lower the SFE, the wider the partial dislocation separation – i.e. the wider the stacking fault (Kelly et al., 2000, Schoeck, 1999).

2.1.1 Calculations of SFE

Different approaches to determine the SFE of metals are available in the literature. The first calculations of the SFE in FCC metals were based on the electron theory of metals (Schoeck and Seeger, 1955). Later, Cotterill and Doyama (Cotterill and Doyama, 1966) calculated the SFE of copper by a variational method using a central-force approximation, where the pairwise interaction between discrete atoms was represented by a Morse potential function. Olson and Cohen (Olson and Cohen, 1976) proposed a thermodynamic modeling approach to calculate the SFE. With the extended node method, Whelan (Whelan, 1959) presented an experimental approach to measure the SFE based on electron diffraction measurements using Transmission Electron Microscopy (TEM). Line profile analysis of XRD spectra is another experimental technique which can be used to evaluate the SFE (Talonen and Hänninen, 2007).

The formation of a stacking fault produces an interface separating two regions of the matrix, as illustrated in Figure 2-1. Due to the change in chemical bonding, the stacking fault can be treated as a specific case of a Gibbs interface (Ferreira and Müllner, 1998). Alternatively, the stacking fault can be treated as a separate phase with stacking variations and two interface boundaries parallel to the fault plane, as illustrated in Figure 2-2. The interfaces result from a difference in the layer spacing between the matrix and the stacking fault phase (SFP) (Hirth and Cohen, 1970).

	2
	\triangleright
Matrix D D	\triangleright
	\triangleright
	\triangleright
	\triangleright
	\triangleright
Matrix \triangleright \triangleright \triangleright	\triangleright
	\triangleright

Figure 2-1: The stacking fault is assumed to be an interface separating two rigidly displaced matrix areas; (a) infinite stacking fault illustrated by stacking operators; (b) terminated stacking fault bounded by two partial dislocations (Ferreira and Müllner, 1998).



Figure 2-2: The stacking fault is considered as a separate phase consisting of two layers with a different stacking sequence; (a) infinite stacking fault; (b) terminated stacking fault (Ferreira and Müllner, 1998).

The thermodynamic approach to calculating the stacking fault energy, which will be used throughout this thesis, can be summarized as follows following the method of Olsen and Cohen (Olson and Cohen, 1976). After introducing a stacking fault into the Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering. undisturbed stacking sequence of a perfect crystal, a change in the Gibbs energy (ΔG) will occur, the magnitude of which must be the same regardless of the approach, i.e., the interface approach or volume approach:

$$\Delta G_{if} = \Delta G_{vol} \tag{2-3}$$

where ΔG_{if} and ΔG_{vol} are the change in Gibbs energy in the case of the interface approach and volume approach respectively. ΔG_{if} can be expressed as

$$\Delta G_{if} = A \Gamma^{\infty} \tag{2-4}$$

where *A* is the interfacial area and Γ^{∞} the interfacial energy per unit area of interface. The change in the Gibbs energy based on the volume approach, on the other hand, can be expressed as

$$\Delta G_{vol} = V^{SFP} (G_V^{SFP} - G_V^M) + 2A\sigma$$
(2-5)

where V^{SFP} is the volume of the SFP, G_V^{SFP} the Gibbs energy per unit volume of SFP, G_V^M the Gibbs energy per unit volume of matrix, and σ the interfacial energy per unit area of phase boundary. By substituting Eq. 2-4 and 2-5 into Eq. 2-3 and differentiating Eq. 2-3 with respect to A

$$\frac{\partial \Delta G_{if}}{\partial A} = \frac{\partial \Delta G_{vol}}{\partial A}$$
(2-6)

one gets an expression for the ideal stacking fault energy:

McMaster – Materials Science & Engineering.

$$\Gamma^{\infty} = 2s(G_V^{SFP} - G_V^M) + 2\sigma \tag{2-7}$$

where s is the interplanar spacing between close-packed planes parallel to the fault plane. In most cases, it is more convenient to work with molar Gibbs energies. The ideal stacking fault energy is then expressed as:

$$\Gamma_{SFE} = 2\rho\Delta G^{\gamma \to \varepsilon} + 2\sigma \tag{2-8}$$

with ρ , the molar surface density of the FCC {111} planes and $\Delta G^{\gamma \to \varepsilon}$, the molar Gibbs energy of the γ (FCC) $\rightarrow \varepsilon$ (HCP) phase transformation. The molar surface density ρ can be calculated by using the lattice parameter *a* and Avogadro's constant *N* as

$$\rho = \frac{4}{\sqrt{3}} \frac{1}{a^2 N} \tag{2-9}$$

Strictly speaking, one has to distinguish between infinite stacking faults and terminated stacking faults, which are associated with Γ^{∞} and Γ^* respectively (Ferreira and Müllner, 1998, Muèllner and Ferreira, 1996). The above derivations used the ideal SFE Γ^{∞} , which is defined as the energy per unit area of an infinitely extended stacking fault in an infinite ideal crystal. Due to the additional energy associated with the strain field, the effective SFE Γ^* is $2s\Delta G_s$ higher than Γ^{∞} , i.e.,

$$\Gamma^* = 2\rho\Delta G^{\gamma \to \varepsilon} + 2\sigma + 2s\Delta G_s \tag{2-10}$$

where ΔG_s is the strain energy per unit volume of the stacking fault. The strain energy term ΔG_s is shape dependent, i.e., sensitive to dislocation configuration, the presence of

nearby defects, grain size, etc. (Khachaturyan, 2008). Thus, Γ^* is not an intrinsic material property as opposed to Γ^{∞} . Muèllner and Ferreira (Muèllner and Ferreira, 1996) have estimated ΔG_s for the particular case of two parallel dislocations as

$$\Delta G_s = \frac{\pi D \varepsilon^2}{2} = \frac{\mu \varepsilon^2}{4(1-\upsilon)} \tag{2-11}$$

where v the Poisson's ratio, and ε the strain normal to the fault plane.

2.1.2 Factors Affecting SFE

An alloy's SFE mainly changes with composition and temperature. Since the SFE is proportional to the Gibbs energy change for the γ (FCC) $\rightarrow \varepsilon$ (HCP) phase transformation (see Eq. 2-8), the SFE increases with temperature for a constant chemical composition (Remy and Pineau, 1977). Furthermore, other microstructural parameters, such as grain size (Lee and Choi, 2000) or the local stress and strain (Byun, 2003) have also been reported to affect the SFE. Lee and Choi (Lee and Choi, 2000) suggested that with decreasing austenite grain size, especially below $\approx 30 \,\mu\text{m}$, the SFE in high-Mn steels increases. They explained that dislocation dissociation might be affected by internal stresses, whose magnitude depends on the austenite grain size. However, they did not discuss the nature of the internal stresses in detail. Moreover, it should be noted that stacking fault size is at the nano-scale whereas grain size is at the micro-scale. Thus, making a correlation between these two seems debatable.

Manganese has only a small effect on solid solution strengthening in high-Mn steels but has a strong effect on the SFE. According to Jiang et al. (Jiang et al., 1998), Mn

McMaster – Materials Science & Engineering.

decreases the SFE in Fe-Mn-Si alloys. Gebhardt et al. (Gebhardt et al., 2010), on the other hand, reported an increase of the SFE with the Mn content in Fe-Mn system. Some of the discrepancies may be explained by the fact that the effect of Mn on the SFE depends on the initial Mn content of the alloy. This is illustrated in Figure 2-3, which shows the influence of Mn on the SFE of austenite in binary Fe-Mn alloys according to Lee and Choi (Lee and Choi, 2000), to Schumann (Schumann, 1974), and to Volosevich et al. (Volosevich et al., 1976). The data collected by these investigators have the same trends: at low Mn contents, Mn additions lead to a lower SFE, but at high Mn levels, increasing Mn content increases the SFE. Lee and Choi (Lee and Choi, 2000) and Schumann's (Schumann, 1974) results are in good agreement, indicating that a SFE minimum is obtained for Mn contents in the range of 13 to 14 at. % Mn. These results differ from the minimum location reported by Volosevich et al. (Volosevich et al., 1976). Adeyev and Petrov (Adeyev and Petrov, 1979) explained that such a minimum is correlated to a saturation of solute segregation at crystal imperfections. Kim et al. (Kim et al., 2009), on the other hand, found by a modified embedded-atom method that segregation does not cause an appreciable effect, while the presence of the minimum was predicted. Lee and Choi (Lee and Choi, 2000) attributed such a minimum to the presence of a minimum for $\Delta G^{\gamma \to \varepsilon}$ (in Eq. 2-8).



Figure 2-3: Variation of SFE as a function of Mn content in Fe-Mn alloys (Lee and Choi, 2000, Schumann, 1974, Volosevich et al., 1976).

Carbon is considered as an effective austenite stabilizer and is usually added to modern high manganese steels in levels up to 0.6 wt. %. Carbon contents higher than this amount make high-Mn steels difficult to process and also lead to poor weldability (Mendez et al., 2004). The influence of carbon content on SFE has been investigated by several authors (Abbasi et al., 2011, Allain et al., 2004b, Brofman and Ansell, 1978, Dumay et al., 2008, Gholizadeh et al., 2013, Medvedeva et al., 2014, Nakano and Jacques, 2010, Petrov, 1993, Saeed-Akbari et al., 2009, Schramm and Reed, 1975). Schramm and Reed (Schramm and Reed, 1975) experimentally determined the SFE of several austenitic stainless steels with carbon contents up to 0.91 wt.%. They found a linear increase of SFE with an addition of carbon. On the other hand, Brofman and Ansell (Brofman and Ansell, 1978) reported that SFE was relatively insensitive to carbon amount in austenitic stainless steel with a maximum of 0.29 wt.% carbon content.
According to the experimental work of Petrov (Petrov, 1993), carbon decreased the SFE of austenitic steels at low concentrations but increased it at high concentrations. These controversies in the experimental work resulted in theoretical studies for SFE calculations. For example, several models have been proposed for the prediction of SFE for Fe-Mn-C system (Allain et al., 2004b, Dumay et al., 2008, Nakano and Jacques, 2010, Saeed-Akbari et al., 2009). According to these models, the SFE increases with increasing carbon content. In recent theoretical works, ab initio simulations have predicted a significant increase of SFE with carbon content (Abbasi et al., 2011, Gholizadeh et al., 2013, Medvedeva et al., 2014).

The addition of aluminum to high manganese steels considerably increases the SFE, e.g. $\approx 5 \text{ mJ/m}^2$ per wt. % Al (Dumay et al., 2008, Hamada et al., 2007). Moreover, the high Al content of this steel class is remarkably advantageous for weight reduction. Silicon strengthens austenite owing to solid solution hardening. In the Fe-Mn-C system, Si has been reported to increase the SFE at low concentrations (<4wt.%), but decreases it at high concentrations (> 4wt. %) (Dumay et al., 2008). However, Takaki et al. (Takaki et al., 1990) found that the addition of 2 wt. % Si to an Fe-27Mn steel lowered the SFE of austenite.

2.1.3 SFE Dependence of Deformation Products

The SFE value has a major influence on the deformation products in high-Mn steels. Generally, with a reduction in SFE, plastic deformation is achieved by: (i) glide of partial and perfect dislocations, (ii) dislocations glide and mechanical twinning, and (iii)

dislocations glide and mechanical ε-martensitic transformation (Allain et al., 2004a, Allain et al., 2004b, Bouaziz and Guelton, 2001, Lee and Choi, 2000, Nakano and Jacques, 2010, Remy and Pineau, 1977, Saeed-Akbari et al., 2009, Sato et al., 1989). Several models have been proposed for the relationship between the alloy SFE and deformation products in high-Mn steels (Allain et al., 2004b, Dumay et al., 2008, Nakano and Jacques, 2010, Remy and Pineau, 1977, Saeed-Akbari et al., 2009).

Allain et al. (Allain et al., 2004b) proposed a model for the calculation of the SFE in Fe-Mn-C austenitic alloys at different temperatures. According to their model, the $\gamma \rightarrow \epsilon$ mechanical martensitic transformation can only take place if the SFE was lower than 18 mJ/m² whereas mechanical twins formed when the SFE lay between 12 and 35 mJ/m². The plot of their calculated SFE values versus temperature for an Fe-22Mn-0.6C steel is presented in Figure 2-4. The magnetic and chemical contribution changes with temperature are also plotted. It can be seen that the SFE increased from 10 mJ/m² at 77K to 19 mJ/m² at 293 K (Allain et al., 2004b).



Figure 2-4: Calculated SFE as a function of temperature for Fe-22Mn-0.6C steel (Allain et al., 2004b).

The Allain et al. SFE model (Allain et al., 2004b) was then extended by Dumay et al. (Dumay et al., 2008) to Fe-Mn-C alloys containing Cu, Cr, Al and Si. Comparison of SFE values as a function of temperature for a Fe-22Mn-0.6C alloy derived from the Allain et al. and Dumay et al. SFE models (Allain et al., 2004b, Dumay et al., 2008) are presented in Figure 2-5. Comparing Figure 2-5 with Figure 2-4, it can be seen that at low temperatures (below 293 K), the predicted SFE values from the Allain et al. model (Figure 2-5) are higher than those presented in their paper (Figure 2-4). For the Dumay et al. SFE model (Figure 2-5), it can be seen that the SFE value for a Fe-22Mn-0.6C alloy decreased from 21 mJ/m² at 293 K to 7 mJ/m² at 77 K. These SFE values are consistent with the deformation products that were detected for this alloy (Allain et al., 2004b).



Figure 2-5: The SFE values as a function of temperature for Fe-22Mn-0.6C alloy; data was derived using Allain et al. and Dumay et al. SFE models (Allain et al., 2004b, Dumay et al., 2008).

In another study, Saeed-Akbari et al. (Saeed-Akbari et al., 2009) used a subregular solution thermodynamic model to calculate the SFE of high-Mn steels. According to their model, the $\gamma \rightarrow \epsilon$ mechanical martensitic transformation took place if the SFE was lower than 20 mJ/m². However, this model does not provide any SFE value range for mechanical twinning.

Nakano and Jacques (Nakano and Jacques, 2010) re-examined the thermodynamic parameters of the ε-martensite phase with respect to austenite in the Fe-Mn system. Based on their proposed parameters, they calculated the SFE in the Fe-Mn and Fe-Mn-C systems as a function of temperature, Mn and C levels. Similar to the model proposed by Saeed-Akbari et al. (Saeed-Akbari et al., 2009), this model does not provide any SFE predicts a weaker effect of carbon content on SFE value.

2.2 Twinning

As was mentioned earlier, twins appear within the microstructure of some high-Mn steels with specific SFE values as a consequence of plastic deformation. They act as obstacles for the movement of gliding dislocations, leading to increase in the work hardening rates of the steels. Thus, it seems necessary to understand the basic aspects of the twins. A twin is a structure whose crystal lattice is a mirror image of that of its parent (Callister and Rethwisch, 2012). Twins are commonly formed during solidification, recrystallization, solid-state phase transformation and plastic deformation. Mechanical twins (or deformation twins) are created as a consequence of a homogeneous shear in the parent lattice so that the product has the same crystal structure as the parent, but has a defined orientation relationship (Kelly et al., 2000). In the case of FCC metals, the twinning elements are: {111} as the twinning plane and $<11\overline{2} >$ as the twinning direction (Qin and Bhadeshia, 2008). Idrissi et al. (Idrissi et al., 2010) found the same twinning elements for an Fe-20Mn-1.2C alloy by TEM.

The morphology of deformation twins in high-Mn steels are very different from either annealing twins or deformation twins observed in other metallic crystal structures (e.g. HCP metals). Under a light microscope, twins of high-Mn steels appear as thin bands of a few tens of micrometers width. However, at a much smaller scale, provided by Ph.D. Thesis – M. Ghasri Khouzani McMaster

McMaster – Materials Science & Engineering.

transmission electron microscopy (TEM), twin bands appear to be stacks of microtwins having thicknesses ranging from a few nanometers to several micrometers depending on the degree of plastic strain (Allain et al., 2004c). A schematic representation of the clustering of microtwins into stacks in a Fe-22Mn-0.6C alloy is illustrated in Figure 2-6. Barbier et al. (Barbier et al., 2009) reported that the twin stacks became thicker at higher strains in the case of the Fe-22Mn-0.6C TWIP steel. By contrast, Gutierrez-Urrutia and Raabe (Gutierrez-Urrutia and Raabe, 2011) observed the decrease in the thickness of the twin stacks with increasing true strain in the same TWIP steel. This discrepancy can be explained by the difference in characterization technique they used, where the EBSD may not have differentiated the austenite matrix between twin stacks, leading to overestimation of twin stack thickness (Barbier et al., 2009). Electron channeling contrast imaging (ECCI) technique applied by Gutierrez-Urrutia and Raabe, 2011) for twin observation, however, has a better angular resolution compared to EBSD.



Figure 2-6: Clustering of microtwins into stacks (Allain et al., 2004c).

2.3 Martensitic Transformation

In high-Mn steels with low SFE, martensitic transformations take place thermally and mechanically. It is necessary to understand these transformations well because they have a major influence on microstructure and mechanical behaviour of low-SFE high-Mn alloys. Martensitic transformations that take place spontaneously during cooling are known as thermal martensite. In the Fe-Mn system, thermal martensitic transformation may be athermal or isothermal. An athermal transformation starts abruptly at a defined, martensite start (M_S), temperature and does not show any time dependence. In an isothermal transformation, however, the M_S temperature is not well-defined and the transformation takes place within a narrow temperature range without a specific incubation time (Sahu and De, 2002). Olson and Cohen (Olson and Cohen, 1972) proposed a classification for martensitic transformations in ferrous systems, shown in Figure 2-7. It should be noted that M_{s}^{σ} is the stress induced martensite temperature and M_d the deformation induced martensite temperature. At temperatures below M_s , martensite forms spontaneously (thermally activated transformation). In the temperature range above M_S and below M_S^{σ} , the martensitic transformation can take place if the summation of mechanical energy due to an externally applied stress and the chemical driving force exceeds a critical activation energy. The transformation is known to be "stress-assisted". In the temperature regime between the M_S^{σ} and M_d , when the externally applied stress exceeds the yield stress of the parent austenite, "strain-assisted" martensite forms.



Figure 2-7: Schematic of the different forms of martensitic transformations (Olson and Cohen, 1972).

Studies on martensitic transformations in Fe-Mn-C alloys have indicated that austenite can be transformed to both $\alpha'(BCT)$ and ϵ (HCP) martensites and that the $\epsilon \rightarrow \alpha'$ transformation is also possible (Grässel et al., 2000). Formation of these phases depends on the chemical composition, the test temperature and the applied strain (Grässel et al., 2000, Liang, 2008, Liang et al., 2009). Schumann (Schumann, 1967) investigated the phases present in un-deformed Fe-Mn alloys by X-ray diffraction (XRD), dilatometry and microstructural characterization. His results can be summarized as follows:

- (i) if the alloy Mn content was less than 15 wt.%, only the $\gamma \rightarrow \alpha'$ transformation took place;
- (ii) when the Mn level was between 15 and 27 wt.%, the microstructure contained both ε martensite and austenite;
- (iii) in alloys with Mn contents greater than 27 wt.%, the microstructure was fully austenitic.

20

In the case of Fe-Mn-C alloys, it should be noted that carbon can prevent the thermal martensite formation within the microstructure by decreasing M_S . For example, Yang (Yang, 2010) showed that the increase in carbon content from 0.2 wt. % to 0.4 wt. % resulted in the change in un-deformed microstructure from a dual-phase microstructure of austenite and ε -martensite to fully austenitic for Fe-22Mn-C alloys. Similarly, adding 0.6 wt. % C to Fe-18Mn-0C alloy suppressed the formation of the thermal ε -martensite, leading to a fully austenitic microstructure at room temperature (Kim et al., 2013). The carbon concentration dependence of transformation temperatures in Fe-17Mn-C steels is depicted in Figure 2-8 (Koyama et al., 2011). It should be noted that A_S is the thermally induced starting temperature of the ε -martensite to austenite transformation. From Figure 2-8, it can be seen that both the M_S and A_S decreased linearly with increasing carbon concentration. It can also be seen that for the alloy carbon content higher than approximately 0.38 wt. %, M_S is lower than 293 K, indicating that thermal ε -martensite cannot form at room temperature.



Figure 2-8: Transformation temperatures as a function of carbon concentration in Fe-17Mn-C steels (Koyama et al., 2011).

The transformation from FCC austenite to HCP ε -martensite is known to be fully coherent (Christian, 2002), because the closely-packed planes and directions in the two structures are parallel. i.e. $\{111\}_{\gamma}$ // $\{0001\}_{\varepsilon}$ and $\langle 1\overline{10} \rangle_{\gamma}$ // $\langle 1\overline{2}10 \rangle_{\varepsilon}$. On the other hand, the orientation relationship between FCC austenite and BCT α '-martensite is usually described by the Kurdjumov-Sachs (K-S) relation. The standard variant of this (Bracke et al., 2006) is given by $\{111\}_{\gamma}$ // $\{101\}_{\alpha'}$ and $\langle 1\overline{10} \rangle_{\gamma}$ // $\langle 11\overline{1} \rangle_{\alpha'}$. It is also worth noting that α' -martensite forms at the intersection of different ε -martensite variants or directly from the austenite phase (Liang et al., 2009, Nihei et al., 2004, Yang and

Wayman, 1992). It is believed that deformation twins and stacking faults act as nucleation sites for formation of α' -martensite (Nihei et al., 2004).

Fujita and Ueda (Fujita and Ueda, 1972) studied the formation mechanism of deformation-induced HCP martensite in an AISI 304 stainless steel by TEM. Their observations indicated that the nucleation of HCP ε -martensite is based on the dislocation dissociation into the partial dislocations a/6 <211> on alternate {111} planes (see Eq. 2.1). The motion of such partial dislocations on every second close-packed plane produces an intrinsic stacking fault with an HCP crystal structure (Olson and Cohen, 1976). This is schematically illustrated in Figure 2-9. In a similar study, Venables (Venables, 1962) compared the thickness of an ε -platelet with the number of partial dislocations on its interface in an AISI 304 stainless steel. It was found that the overlapping process of stacking faults occurs in a regular manner. Most of the faults nucleate from the following sites (Fujita and Ueda, 1972): (i) grain boundaries and annealing twin boundaries; (ii) intersections between different families of slip planes.

	1 partial	2 partials	3 partials	S
A	F	4	A	A
В	1 E	З	В	в
С		4	A	A
А	E	3 1	В	B
В	\rightarrow (℃	A	A Hep
С	\longrightarrow	A→	BL	B
А	I → E	3 →	C _ →	A
В	\rightarrow (⊃ —→	A →	В
С	→ <i>4</i>	A →	В →	C

Figure 2-9: Schematic illustration of nucleation mechanism for ε martensite (Bracke et al., 2006).

Putaux and Chevalier (Putaux and Chevalier, 1996) have shown that thermallyinduced $\gamma \rightarrow \varepsilon$ transformation occurs by the correlated glide of partial dislocations on every second {111} plane in an Fe-Mn-Si-Cr-Ni shape memory alloy. They also observed that a large ε -martensite plate is composed of thinner ε -martensite layers having a thickness of a few to a few dozen atomic planes and separated by retained austenite layers. Figure 2-10 presents a high-resolution TEM micrograph of such an ε -martensite plate. Similar observations were reported by Liang (Liang, 2008) for a Fe-24Mn-0C alloy and by Yang (Yang, 2010) for Fe-22Mn-0.2C and Fe-22Mn-0.4C alloys.



Figure 2-10: High resolution image showing the layered substructure of a large ε -martensite plate (Putaux and Chevalier, 1996).

2.4 Strain Hardening

The exceptional mechanical properties of high-Mn steels are a consequence of their high strain hardening rates which sustain at high strains. The role of deformation products in enhancing the mechanical properties can also be quantified by understanding the strain hardening behaviour of these steels. For this reason, the fundamental aspects of the strain hardening of high-Mn steels are reviewed in this section. In plasticity theory, there are two types of strain hardening: isotropic and kinematic strain hardening. For isotropic strain hardening, the relationship between the increase in flow strength and equivalent strain does not depend on the stress state: i.e. the initial yield surface expands uniformly during plastic flow (Bate and Wilson, 1986). In the case of kinematic strain hardening, however, the yield surface does not change in its initial form and orientation but translates in stress space like a rigid body. Kocks and Mecking (Kocks and Mecking, 2003) proposed that kinematic strain hardening is not an alternative to isotropic strain Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering. hardening, but can be present a second state parameter. In more recent studies, Bouaziz et al. (Bouaziz et al., 2008) and Yang (Yang, 2010) found a strong contribution of kinematic strain hardening to the mechanical behaviour of an Fe-22Mn-0.6C TWIP alloy.

Three stages of isotropic strain hardening are observed in the strain hardening of all FCC single crystals. Stage I, or easy glide, is characterized by a low rate of strain hardening. Stage II, or the linear hardening region, exhibits a rapid increase in strain hardening rate. Stage III, or the parabolic hardening region, the onset of which is noticeably dependent on temperature, shows a lower rate of strain hardening (Kocks and Mecking, 2003). In polycrystals, stage I is usually not observed and quite typically stage II is masked by the distribution of friction stresses and the differing Schmid/Taylor factors among the grains (Bouaziz et al., 2008). The Kocks-Mecking model essentially concerns stage III of strain hardening. The core concept of this model is that the evolution of dislocation density accounts for dislocation accumulation in the material due to forest interactions, and dynamic recovery due to dislocation annihilation and cross-slip. These two processes can be described mathematically by the following equation (Kocks and Mecking, 2003):

$$\Theta = \Theta_0 \left(1 - \frac{1}{\mu} \left(\frac{\sigma_{V0}}{\mu_0}\right)^{-1} \frac{1}{r(\dot{\varepsilon}, \mathrm{T})} \sigma\right)$$
(2-12)

where Θ is the work hardening rate due to dislocation glide, Θ_0 the athermal work hardening rate, μ the shear modulus of the material, $\frac{\sigma_{V0}}{\mu_0}$ the ratio of scaling stress to Ph.D. Thesis – M. Ghasri KhouzaniMcMaster – Materials Science & Engineering.shear modulus at 0K and σ the flow stress. The last term in Eq. 2-12 reflects thecontribution of dynamic recovery (Kocks and Mecking, 2003).

Several plots have been developed to correlate the scaling stress and material SFE, one of which is displayed in Figure 2-11. For all plots, a small hump can be observed in the middle part which has been attributed to the strain aging due to impurities. The two border lines for the SFE values of zero and infinity were also extrapolated (Kocks and Mecking, 2003). The $\frac{\sigma_{V0}}{\mu_0}$ ratio in Eq. 2-12 for FCC metals can be found using Figure

2-11.



Figure 2-11: $(\sigma_V/\mu)^{1/2}$ versus $\left[\frac{kT}{\mu b^3}Ln(\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}})\right]^{1/2}$ plots for some FCC metals. χ is the stacking

fault energy (SFE) value (Kocks and Mecking, 2003).

The $r(\dot{\varepsilon},T)$ term in Eq. 2-12 can be defined by following expression (Kocks and Mecking, 2003):

$$r(\dot{\varepsilon},T) = \left(\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}}\right)^{kT/A}$$
(2-13)

where $\dot{\varepsilon}$ is the applied strain rate, *T* the test temperature, $\dot{\varepsilon}_0$ a fitting parameter, *k* the Boltzmann constant and *A* a parameter dependent on the material SFE. In the Kocks-Mecking approach, the correlation between the *A* parameter and material SFE was not defined. However, Liang et al. (Liang, 2008, Liang et al., 2009) developed the following correlation equation for a low C Fe-24Mn steel:

$$kT / A = C \cdot \frac{T}{T_M} \cdot \chi^n \tag{2-14}$$

where χ is the material SFE, T_M the material melting point, and *C* and *n* are fitting parameters. Substituting Eq. 2-13 and Eq. 2-14 into Eq. 2-12, Liang et al. (Liang, 2008, Liang et al., 2009) defined the work hardening rate due to dislocation glide as a function of SFE as:

$$\Theta = \Theta_0 \left(1 - \frac{1}{\mu} \left(\frac{\sigma_{V0}}{\mu_0}\right)^{-1} \left(\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}}\right)^{-C \frac{T}{T_M} \chi^n} \sigma\right)$$
(2-15)

2.5 High Manganese TWIP Steels

High manganese TWIP steels are a type of transformable steels which exhibit high strength and large ductility owing to their excellent strain hardening characteristics. This

makes these steels an attractive candidate for the automobile industry. The origin of the high strain hardening rate of TWIP steels have been a subject of debate. Numerous authors attributed this to mechanical twinning, where created twin boundaries effectively decrease the mean free path for dislocation motion leading to a "dynamic Hall-Petch effect" (Allain et al., 2002, Allain et al., 2004a, Allain et al., 2004b, Barbier et al., 2009, Bouaziz et al., 2008, Bouaziz and Guelton, 2001, Bouaziz et al., 2011a, Gutierrez-Urrutia and Raabe, 2012, Gutierrez-Urrutia et al., 2010, Remy, 1978). On the other hand, some authors have advocated the hypothesis that the high strain hardening rates in high-Mn TWIP steels was a result of the dynamic strain aging (DSA) effect (Adler et al., 1986, Dastur and Leslie, 1981). The details of these two effects are presented below.

2.5.1 Dynamic Hall-Petch Effect

In TWIP steels, mechanical twins do not thicken significantly with deformation level. However, the twin volume fraction increases through nucleation of new deformation twins (e.g. activation of secondary twinning systems). As deformation proceeds, grains are continuously sub-divided by twin boundaries, which is often called dynamic Hall-Petch effect as this effectively reduces the dislocation mean free path with increasing deformation. Such a dynamic grain refinement is schematically illustrated in Figure 2-12. The role of twins on the strain hardening rate of TWIP steels has also been quantified by several authors (Bouaziz et al., 2008, Bouaziz and Guelton, 2001, Karaman et al., 2001, Remy, 1978).



Figure 2-12: Schematic illustration of dynamic Hall-Petch effect (De Cooman et al., 2009).

2.5.2 Dynamic Strain Aging (DSA) Effect

The uniaxial tensile curves of high manganese TWIP steels with more than 0.4 wt. % C often exhibit serrations (see Figure 2-13). This serrated flow, which is also called "jerky flow" and is often referred to as the "Portevin-LeChatelier (PLC) effect", is usually attributed to dynamic strain aging (DSA) and negative strain rate sensitivity (Chen et al., 2007, Renard et al., 2010). The PLC effect has not been observed in high manganese steels whose carbon level is less than 0.1 wt. % (Grässel et al., 2000, Liang et al., 2009). For this reason, the DSA effect is usually described as originating through interactions between carbon atoms and dislocations. When the mobility of carbon atoms and velocity of dislocations are similar to each other, dislocations can be locked by carbon atoms. In the case of plain carbon steels, the DSA effect is usually observed when the test temperature is higher than 373 K. For TWIP steels, however, this phenomenon is often observed at room temperature (Figure 2-13).



Figure 2-13: Serrations in tensile curve of Fe-20Mn-1.2C TWIP steel tested at room temperature (Renard et al., 2010).

Dastur and Leslie (Dastur and Leslie, 1981) proposed another model for describing the DSA phenomenon in high manganese steels (see Figure 2-14). According to this model, C-Mn pairs lock the dislocations by reorienting themselves in the stress field at the dislocation core. They also suggested the DSA as the main cause of work hardening in high-Mn alloys. In a recent study, however, Kim et al. (Kim et al., 2013) reported that the contribution of DSA to the flow stress of Fe-18Mn-0.6C and Fe-18Mn-0.6C-1.5Al TWIP steels was less than 20 MPa. Based on EBSD and TEM analyses, they attributed the high work hardening rates of their TWIP steels to the dynamic Hall-Petch effect.



Figure 2-14: Dastur-Leslie model for DSA in Hadfield steels (De Cooman et al., 2009).

2.5.3 Work Hardening Behaviour

High manganese TWIP steels with various chemical compositions generally exhibit the same work hardening behaviour (Chen et al., 2007, Barbier et al., 2009, Renard et al., 2010, Renard and Jacques, 2012, Yang et al., 2010). The work hardening behaviour of a Fe-20Mn-1.2C TWIP steel from the study of Renard and Jacques (Renard and Jacques, 2012) as a typical example is presented in Figure 2-15. The work hardening rate curve can be divided to three stages. In stage A, the work hardening rate decreases with increasing strain. In stage B, the work hardening rate increases until it attains a maximum. In part C, the work hardening rate declines until fracture. For a Fe-22Mn-0.6C TWIP steel, the transition between stages A and B was around $\varepsilon = 0.09$ whereas the transition between stages B and C was approximately $\varepsilon = 0.34$, as shown by Yang et al. (Yang et al., 2010).



Figure 2-15: Work hardening rate as a function of true strain for Fe-20Mn-1.2C TWIP steel tensile tested at room temperature; G is the shear modulus (Renard and Jacques, 2012).

The work hardening behaviour of high manganese TWIP steels can be explained by understanding the kinetics of mechanical twinning. Using a point counting method developed by the authors, Renard and Jacques (Renard and Jacques, 2012) estimated the twin volume fraction at different strain levels from EBSD maps. The evolution of twin volume fraction with true strain for their Fe-20Mn-1.2C TWIP steel is depicted in Figure

McMaster – Materials Science & Engineering.

2-16 (Renard and Jacques, 2012). The vertical lines which delimit the three stages in work hardening plot are superimposed in twinning kinetics curve (Figure 2-16). For $\varepsilon < 0.12$, a very small increase in twin volume fraction was observed (Figure 2-16). This corresponded to stage A of the work hardening rate plot (Figure 2-15), where the work hardening rate decreased with increasing strain. This is the typical work hardening behaviour of an FCC metal for which dislocation glide is the dominant deformation mode (Kocks and Mecking, 2003, Liang et al., 2009). At $\varepsilon = 0.12$, the twin volume fraction then increased significantly until $\varepsilon = 0.3$, corresponding to an increase in work hardening rate in stage B. Finally, the twin volume fraction plot saturated at high strains until necking, which corresponded to the decrease in work hardening rate observed in stage C.



Figure 2-16: Twin volume fraction as a function of true strain for Fe-20Mn-1.2C TWIP steel (Renard and Jacques, 2012).

Lu et al. (Lu et al., 2014) investigated the evolution of mechanical twinning for an Fe-22Mn-0.6C TWIP steel subjected to uniaxial tension (UT) and plane strain tension (PST) strain paths. The saturated volume fraction of mechanical twins at fracture was reported to be 0.35 and 0.38 for the UT and PST strain paths, respectively (Lu et al., 2014). This is consistent with the volume fraction of twins at fracture for the Fe-20Mn-1.2C steel which can be inferred to be approximately 0.36 from Figure 2-16 (Renard and Jacques, 2012).

2.5.4 Effect of Chemical Composition on Tensile Properties

The role of SFE on deformation products and work hardening of high manganese steels was previously discussed. However, in the case of high manganese steels with similar SFE values, the mechanical properties change with chemical composition (Bouaziz et al., 2011b). The effect of carbon and manganese content on the yield stress of TWIP steels was quantified by Bouaziz et al. (Bouaziz et al., 2011b) as:

$$YS(MPa) = 228 + 187C - 2Mn \tag{2-16}$$

where *C* and *Mn* are carbon and manganese level of TWIP steels in weight percent. The strong effect of carbon on increasing the yield stress can be explained by its solid solution hardening effect. However, the negative contribution of manganese to yield stress was attributed to the complex interactions between dislocations and C and Mn (Bouaziz et al., 2011b).

Flow curves of several high manganese TWIP steels are presented in Figure 2-17. It should be noted that Fe-30Mn alloy is not a TWIP alloy (Liang et al., 2009), and its tensile behaviour was used in the figure as reference dislocation glide dominated material (Bouaziz et al., 2011b). It can be seen that the work hardening rate of all the TWIP steels was significantly higher than the Fe-30Mn alloy. This was attributed to the role of twinning in decreasing the mean free path for dislocation motion (Bouaziz et al., 2011b). The elongation of all TWIP steels studied seemed to be approximately identical, and was significantly higher than that of the Fe-30Mn alloy. Moreover, Fe-12Mn-1.2C alloy exhibited higher yield stress than Fe-22Mn-1.2C alloy, which had a higher yield stress

than Fe-22Mn-0.6C alloy. This was consistent with the mathematical expression for yield stress provided in Eq. 2-16.



Figure 2-17: True stress-true strain curves of several high manganese TWIP steels as well as Fe-30 Mn alloy (as reference material) (Bouaziz et al., 2011b).

2.5.5 Role of Grain Size

The effect of grain size on yield stress of high manganese TWIP steels can be expressed mathematically through the conventional Hall-Petch equation. As an example, Bouaziz et al. (Bouaziz et al., 2011a) proposed the following equation for fully recrystallized Fe-22Mn-0.6C cold rolled sheet material.

$$YS(MPa) = 132 + \frac{449}{\sqrt{d}}$$
(2-17)

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering. where *d* is austenite grain size in μ m. Since the minimum grain size that can be achieved by industrial processes is around 2.5 μ m, the maximum practical yield stress for fully recrystallized Fe-22Mn-0.6C cold rolled sheet is expected to be≈ 450 MPa (Bouaziz et al., 2011a).

Tensile curves for a series of Fe-22Mn-0.6C TWIP steels with different grain sizes are displayed in Figure 2-18. It can be seen that the yield stress and UTS increased with decreasing grain size from 20 μ m to 1 μ m, while elongation decreased. Moreover, the effect of grain size on the YS and UTS became more significant when the grain size was less than 6 μ m.



Figure 2-18: Engineering stress-engineering strain plots of Fe-22Mn-0.6C TWIP steel as a function of grain size (Bouaziz et al., 2011a).

The evolution of the strain onset for twinning (ε_{init}) and its corresponding stress (Stress (ε_{init})) with the grain size for an Fe-22Mn-0.6C steel is presented in Figure 2-19. It

can be seen that strain onset for twinning increased linearly with increased grain size. However, the corresponding stress remained constant because the flow stress decreased with increased grain size, as shown in Figure 2-18 (Bouaziz et al., 2008).



Figure 2-19: Grain size effect on strain and stress of twinning onset (Bouaziz et al., 2008).

2.6 High Manganese TRIP Steels

High manganese TRIP steels are another type of high strength steel, in which FCC austenite transforms to either BCT α '-martensite or HCP ϵ -martensite as a consequence of plastic deformation. The number of studies on these steels in the literature is not as extensive as those on high-Mn TWIP steels. In this section, the mechanical behaviour, microstructural evolution and fracture mechanism of high manganese TRIP steels are reviewed.

2.6.1 Mechanical Behaviour

The tensile behaviour of an Fe-20Mn-3Si-3Al TRIP steel was investigated by Grässel et al. (Grässel et al., 2000). The undeformed microstructure of the steel comprised

austenite (≈ 80 Vol. %) and ϵ -martensite (≈ 20 Vol. %). The room temperature tensile flow of the steel in terms of (σ_{true} - $\sigma_{true,0.05}$) versus true strain is presented in Figure 2-20. It should be noted that $\sigma_{true,0.05}$ is true stress at true strain of 0.05. An increase in work hardening rate (the slope of the plot) can be observed at a true strain of approximately 0.15 (Grässel et al., 2000).



Figure 2-20: True stress increment versus true strain for Fe-20Mn-3Si-3Al TRIP steel (Grässel et al., 2000).

Liang et al. (Liang et al., 2009) studied the tensile behaviour of an Fe-24Mn-0C TRIP steel. The undeformed steel had a dual phase microstructure of austenite (volume fraction of 0.43) and ε -martensite (volume fraction of 0.57). The true stress-true strain curve of the steel is presented in Figure 2-21. An increase in the slope of the curve can be observed when true strain was approximately \approx 0.06. However, the stress increment

declined when the strain reached ≈ 0.2 . Compared to the Fe-20Mn-3Si-3Al TRIP steel of Grässel et al. (Grässel et al., 2000), this steel had a significantly lower elongation.



Figure 2-21: Tensile behaviour of Fe-24Mn-0C alloy at room temperature (Liang, 2008).

The tensile behaviour of an Fe-22Mn-0.2C TRIP steel was investigated by Yang et al. (Yang et al., 2010). The undeformed microstructure of the steel comprised austenite (volume fraction of 0.93) and ε -martensite (volume fraction of 0.07). The room temperature tensile behaviour of the steel was very similar to that observed in Figure 2-21 for Fe-24Mn-0C alloy.

The tensile response of an Fe-17Mn-0.6C steel at 173 K and 223 K is depicted in Figure 2-22 (Koyama et al., 2011). The test temperature of 173 K was lower than the alloy M_S whereas the 223 K was between M_S and M_d (see §2.3). Thus, the undeformed microstructure of the Fe-17Mn-0.6C steel was a combination of austenite and ε -martensite at 173 K and fully austenitic at 223 K. From Figure 2-22, it can be seen that

the work hardening rate increased and the uniform elongation decreased when the test temperature decreased.



Figure 2-22: True stress-true strain curves and corresponding work hardening plots of the Fe-17Mn-0.6C steel at 173 K and 223 K (Koyama et al., 2011).

2.6.2 Microstructural Evolution

The evolution of volume fraction of alloy phases with true strain for an Fe-20Mn-3Si-3Al TRIP steel is displayed in Figure 2-23. The $\gamma \rightarrow \epsilon$ martensite transformation occurred when the true strain increased from 0 to 0.06. Furthermore, $\gamma \rightarrow \alpha'$ martensitic transformation started from true strain of 0.05 and continued until fracture, resulting in the increase in the tensile plot slope observed in Figure 2-20. Moreover, ϵ -martensite transformed to α '-martensite at high strains. The mechanism of this transformation was described by Sato et al. (Sato et al., 1982) for an Fe-30Mn-1Si alloy. The high total elongation value of this steel was attributed to the significant amount of austenite present in undeformed microstructure (Grässel et al., 2000).



Figure 2-23: Volume fraction evolution of present phases with true strain for Fe-20Mn-3Si-3Al TRIP steel (Grässel et al., 2000).

Figure 2-24 displays the evolution of ε -martensite volume fraction with tensile strain for the Fe-24Mn-0C alloy of Liang et al. (Liang, 2008). It can be seen that the ε martensite volume fraction started to increase from a true strain of ≈ 0.07 , where the plot saturated at high strains. This explained the tensile behaviour observed for this steel in Figure 2-21. Dislocation glide and γ martensitic transformation (TRIP effect) were reported as the deformation modes of this steel (Liang et al., 2009). Moreover, the

relatively low elongation of the steel (see Figure 2-21) was attributed to the significant amount of ε -martensite formed during deformation (Liang et al., 2009). This was consistent with the microstructural evolution study of an Fe-22Mn-0.2C TRIP steel (Yang et al., 2010), where the volume fraction of ε -martensite at necking was \approx 0.4, resulting in low uniform elongation (0.32) . As was mentioned above, the undeformed microstructure of Fe-22Mn-0.2C steel contained a significant amount of austenite (volume fraction of 0.93) (Yang et al., 2010). Thus, the presence of the significant amount of austenite within the undeformed microstructure did not lead to a high uniform elongation, indicating that Grässel's explanation (Grässel et al., 2000) for the high elongation of the Fe-20Mn-3Si-3Al TRIP steel was not universal.



Figure 2-24: Evolution of ε-martensite volume fraction with tensile strain for Fe-24Mn-0C TRIP steel (Liang, 2008).

In the study of Koyama et al., mechanical ε-martensite started to appear within the microstructure of a Fe-17Mn-0.6C steel when the tensile strain approached 0.1 (Koyama et al., 2011). The amount of mechanical ε-martensite formed at 173 K was higher than at 223 K, leading to the higher work hardening rate but lower uniform elongation observed for this test temperature, as shown in Figure 2-22 (Koyama et al., 2011). This is consistent with findings of Liang et al. (Liang et al., 2009), where the ε-martensite volume fraction of deformed Fe-24Mn-0C and Fe-30Mn-0C alloys increased with decreasing the deformation temperature to 77 K, resulting in an increase in the work hardening rate but a decrease in the uniform elongation.

As was mentioned above, the $\gamma \rightarrow \varepsilon$ martensitic transformation dominated at lower deformation temperatures, which can be explained by the increased driving force for this transformation (see §2.1.2). At a given deformation temperature, however, the $\gamma \rightarrow \varepsilon$ martensitic transformation can still be controlled by changing the strain rate. Figure 2-25 depicts the ε -martensite volume at fracture as a function of strain rate for an Fe-12Mn-0.6C steel (Lee et al., 2014). It can be seen that the volume fraction of ε -martensite at fracture increased with decreasing strain rate, which was attributed to an increase in the M_S^{σ} temperature (Lee et al., 2014). The total elongation of the Fe-12Mn-0.6C steel decreased with increasing the ε -martensite volume at fracture (Lee et al., 2014), which was consistent with similar studies (Koyama et al., 2011, Liang et al., 2009, Yang et al., 2010).



Figure 2-25: ε-martensite volume at fracture as a function of strain rate for Fe-12Mn-0.6C steel (Lee et al., 2014).

2.6.3 Fracture Mechanism

It is generally accepted that the appearance of mechanical ε -martensite within the microstructure of TRIP steels decreases the uniform elongation (Koyama et al., 2011, Lee et al., 2014, Liang et al., 2009, Takaki et al., 1990, Yang et al., 2010). The fracture behaviour of high manganese TRIP steels has also been analyzed to investigate the role of ε -martensite (Koyama et al., 2011, Liang, 2008, Takaki et al., 1990, Yang, 2010). Takaki et al. (Takaki et al., 1990) observed step-like ridges in the fracture surface of an Fe-27Mn-7Si alloy deformed at 77 K. The same features were observed in the fracture surface of an Fe-17Mn-0.6C steel deformed at 173 K by Koyama et al. (Koyama et al., 2011), as illustrated in Figure 2-26(a). They concluded that brittle fracture was the

dominant fracture mode for their alloy with ε -martensite plates acting as crack initiation sites (Koyama et al., 2011).

Takaki et al. (Takaki et al., 1990) suggested a mechanism for step-like ridge formation, as schematically illustrated in Figure 2-26(b). According to their model, Shockley partial dislocations piled up and generated stress concentrations at the intersection of primary and secondary ε -martensite plates. As a result, tunnel-like voids formed below the secondary ε -martensite plates, as shown in Figure 2-26(b). The coalescence of the tunnel-like voids results in the formation of the observed step-like ridges (Takaki et al., 1990).



Figure 2-26: Fracture surface of Fe-17Mn-0.6C alloy after tensile testing at 173 K (a) (Koyama et al., 2011) and schematic illustration of step-like ridge formation (b) (Takaki et al., 1990).

Liang (Liang, 2008) investigated the fracture behaviour of a Fe-24Mn-0C TRIP alloy after uniaxial tensile testing at room temperature. The fractography results for this

alloy are summarized in Figure 2-27. The fracture surface (Figure 2-27(a)) was reported to be complex, having both ductile tearing features and flat cleavage-like surfaces (Liang, 2008). In the cross-section of the fracture sample (Figure 2-27(b)), cracks nucleated along the interface and micro-voids nucleated at intersection of ε -martensite plates were observed (Liang, 2008). These damage events were concluded to be responsible for the cleavage features observed on the fracture surface (Liang, 2008).



(a) fracture surface

(b) cross-section near the fracture surface

Figure 2-27: SEM fractographs of Fe-24Mn-0C alloy after uniaxial tensile test at 293 K (Liang, 2008).

In another similar study, Yang (Yang, 2010) analyzed the fracture behaviour of a Fe-22Mn-0.2C TRIP steel after tensile testing at room temperature. Micro-voids, microcracks, cleavage facets and ductile tearing features were observed on the fracture surface (Yang, 2010). The damage event was concluded to be void formation at the intersection of ε -martensite plates and the austenite matrix (Yang, 2010).
2.7 High Manganese TWIP/TRIP Steels

For some high manganese steels, mechanical twinning and martensitic transformation occur simultaneously during deformation. An example is a Fe-22Mn-0.4C steel, the microstructural evolution of which was investigated in two different studies (Lü et al., 2010, Yang et al., 2010). In both contributions, a combination of TWIP and TRIP effect was reported as the deformation mode of the steel. However, Yang et al. (Yang et al., 2010) observed a higher population of deformation twins and less mechanical εmartensite than Lü et al. (Lü et al., 2010). This discrepancy can be explained by slight differences in the carbon content of the steels and also the deformation methods. Yang et al. (Yang et al., 2010) used uniaxial tension for the deformation of the steel with 0.40 wt. % C whereas Lü et al. (Lü et al., 2010) employed rolling for deformation of a steel with a carbon content of 0.376 wt. %. The alloy SFE increases with increasing carbon content, resulting in the TWIP effect becoming dominant (Allain et al., 2004b, Dumay et al., 2008). The change in deformation method can change the effective stress and strain, leading to a difference in the relative ratio of the volume fraction of deformation products.

3 OBJECTIVES

The principal objective of this thesis is to investigate the microstructural and mechanical properties evolution of compositionally graded high-Mn steels as a function of uniaxial plastic deformation. To achieve this goal, two high-Mn alloys were chosen as starting materials: Fe-22Mn-0.6C and Fe-30Mn-0.6C. Carbon gradients from 0 wt. % at the surface to 0.6 wt. % at the core were obtained by decarburizing both Fe-22Mn-0.6C and Fe-30Mn-0.6C alloys as schematically illustrated in Figure 3-1.



Figure 3-1: Schematic illustration of carbon gradient within thickness of carbon graded steels.

In order to understand the effect of carbon content on the microstructure and mechanical behaviour of both Fe-22Mn-C and Fe-30Mn-C alloy systems, another objective of this study is to produce and characterize three homogenous alloys with carbon contents of 0, 0.2 and 0.4 wt. % for each system by decarburizing and annealing the as-received alloys. For both homogeneous and carbon graded alloys, the following were examined in detail:

- 1) Microstructure and phase characterization of the as heat-treated alloys.
- 2) Tensile properties at room temperature.
- 3) Fracture mechanism and damage of the alloys.
- Interrupted tensile tests at room temperature to determine the effect of plastic deformation on the microstructure and phase assemblage as a function of strain.
- 5) Formation kinetics of the deformation products and their role in determining the work hardening characteristics of the alloys.

Finally, the tensile behaviour of the homogenous alloys will be modelled by using a modified Kocks-Mecking model such that the contributions of the twinning and ε -martensite formation kinetics to the work hardening rate of the alloys is explicitly taken into account.

4 EXPERIMENTAL METHODS

This chapter reviews the experimental techniques and methods applied in this work. First, materials that were selected for this project are introduced. Then, the experimental apparatus that were used to investigate the microstructure-mechanical property relationships are described. The characterization techniques that were applied to derive the results are also explained.

4.1 Starting Materials

The current thesis is focused on two high-manganese alloy systems: Fe-22Mn-C and Fe-30Mn-C. The starting materials were cold rolled Fe-22Mn-0.6C and Fe-30Mn-0.6C steel sheets with an as-received thickness of 1.5 mm and 1.6 mm, respectively. Both steels were provided through the Resource for the Innovation of Engineered Materials (RIEM) program of CANMETMaterials. The chemical composition of both as-received steels was analyzed for all elements except carbon and sulfur by two techniques:

- Inductively Coupled Plasma Optical Emission Spectroscopy or ICP OES (Vista-Pro, Varian).
- Glow Discharge Optical Emission Spectroscopy or GDOES (GD Profiler, HORIBA Jobin Yvon).

In order to measure the carbon and sulfur contents of the experimental steels a LECO HF-400 carbon and sulfur combustion analyzer was used. The results of chemical composition analysis of both as-received steels are summarized in Table 4-1. The results of GDOES and ICP-OES were in agreement, and their average is listed in the table. No significant change in manganese concentration within the sample depth was observed by GDOES.

Table 4-1. Chemical composition of the as-received steels.

Steel	Mn (wt. %)	Cr (wt. %)	Si (wt. %)	C (wt. %)	S (wt. %)
Fe-22Mn-0.6C	21.76±0.43	0.13 ± 0.03 0.11±0.02	0.14 ± 0.04	0.57	0.01
Te-301vIII-0.0C	30.20±0.00	0.11 ± 0.02	0.08 ± 0.02	0.39	0.01

The microstructure of the both as-received cold rolled steels is illustrated in Figure 4-1. Both the microstructures comprised austenite grains, some of which looked darker due to orientation contrast. The average grain size was $22\pm3 \mu m$ and $31\pm4 \mu m$ for the Fe-22Mn-0.6C and Fe-30Mn-0.6C, respectively.



(a) Fe-22Mn-0.6C

(b) Fe-30Mn-0.6C

Figure 4-1: SEM micrographs from the microstructure of as-received steels.

4.2 Heat Treatments

To investigate the effect of carbon content on the microstructure and mechanical behaviour of the experimental alloys, decarburization heat treatments under a wet hydrogen atmosphere were used to obtain steels with lower carbon contents of approximately 0, 0.2 and 0.4 wt. %. Annealing heat treatments under an argon

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering. atmosphere were also applied to achieve homogenous alloys in terms of carbon distribution with a similar as-annealed grain size. In order to differentiate between the four homogenous alloys within the Fe-22Mn-C and Fe-30Mn-C systems, they will be referred to by their wt. % carbon contents of 0.6C, 0.4C, 0.2C or 0C in the following text.

To examine the effect of imposing a carbon gradient on the microstructure and mechanical properties of the experimental alloys, decarburization heat treatments under different atmospheres were applied. Three carbon graded Fe-22Mn-C alloys (G1, G2, G3) and one carbon graded Fe-30Mn-C alloy were obtained in this way. In the case of the Fe-30Mn-C system, a monolithic alloy carbon distribution was achieved through annealing a Fe-30Mn-0.6C steel to compare the mechanical properties of the graded alloy with the homogenous one.

All decarburization and annealing heat treatments in this work were performed in a horizontal tube furnace at 1000°C followed by oil quenching. Heat-treated samples comprised 102 mm \times 36 mm coupons. The heat treatment parameters for the Fe-22Mn-C and Fe-30Mn-C alloys are summarized in Table 4-2 and Table 4-3, respectively.

Alloy	Decerburization Atmosphere	Decarburization	Annealing (by Ar 99.999
Name	Decarbulization Atmosphere	Time	%) Time
0C	wet H ₂ (5.2 %)+Ar (94.8 %)	7 h	0
0.2C	wet H ₂ (5.2 %)+Ar (94.8 %)	2.5 h	4.5 h
0.4C	wet H ₂ (5.2 %)+Ar (94.8 %)	40 min	6 h + 20 min
0.6C	-	0	7 h
G1	CO ₂ (99.9 %)	4 h	0
G2	wet H ₂ (5.2 %)+Ar (94.8 %)	1 h	0
G3	wet H ₂ (5.2 %)+Ar (94.8 %)	3 h	0

Table 4-2. Heat treatment parameters for Fe-22Mn-C alloys.

Alloy Name	Decarburization (by wet H_2 (5.2 %)+Ar (94.8 %))	Annealing (by Ar 99.999
	Time	%) Time
0C	7 h	0
0.2C	2 h + 50 min	4 h + 10 min
0.4C	50 min	6 h + 10 min
0.6C	0	7 h
Graded	1 h + 15 min	0
Monolithic	0	1 h + 15 min

Table 4-3. Heat treatment parameters for Fe-30Mn-C alloys.

4.3 Sample Preparation

The 102 mm \times 36 mm coupons for heat treatment were cut from the as-received steel sheets by water jet. Tensile samples were machined from the heat-treated coupons using electrical discharge machining (EDM) to prevent any stress or strain-induced transformation and also provided high precision. For microstructural characterization purposes, small samples were cut from as heat-treated coupons or tensile samples using a Struers[®] Accutom-5 precision cut-off saw.

All samples were cold mounted for metallographic examination. Relatively standard metallographic grinding and polishing methods were used in the current work for preparing samples for scanning electron microscopy (SEM) imaging, electron backscattered diffraction (EBSD) observation and X-ray diffraction (XRD) analysis. Since the materials in this work were sensitive to deformation, it was important to carefully control the applied force during grinding and polishing processes. To achieve this, a Struers[®] Rotopol-31 Automatic Polisher was used in an "EBSD Prep" mode. To reduce mechanical damage further, a force of 5 N per sample and rotation speed of 150 rpm was applied.

The samples with more than 0.2 wt. % C were etched with a 5 % nital etchant. A tint etching method was used for the un-deformed steels with very low carbon contents. The tint etching method involved two steps: pre-etching with 5% nital for a few seconds followed by and immersion in Klemm's I etchant (Vander Voort, 1984) until the surface became purple. After etching, all samples were cleaned and rinsed with ethanol and dried.

For EBSD analysis, sample preparation is critical because the diffracted electrons come from the top few tens of nanometers of the sample surface. Ion milling is capable of removing the surface residual strain layer and yielding a scratch free surface, especially when samples posses hard and soft phases. This technique involves bombarding a selected area of the sample surface by an energetic ion beam under vacuum.

In preparing EBSD specimens for the present work, the sample cross sections were initially prepared using the standard metallographic procedures described above followed by Ar ion milling via a JEOL IB-09010 Cross-section Polisher (CP). An acceleration voltage of 6 keV and a current of 165-185 μ A were applied.

4.4 Mechanical Testing

All mechanical testing in this work utilized a 10 kN Instron 5566 universal testing machine and all samples were tested at room temperature (20°C or 293K). Tests included uniaxial tensile testing until fracture and interrupted tensile testing. In all cases, the load was measured by a 10 kN load cell and the sample tensile extension was recorded using a 100 % extensometer with a 25.0 mm gauge length. The cross-head speed was set at 1.0 mm/min for all samples, which approximately corresponded to a 6.67×10^{-4} s⁻¹ strain rate.

Sub-size tensile samples were used for all tensile tests, where the geometry conformed to ASTM E8M (2004). All tensile samples were cut from heat-treated coupons along the rolling direction using electrical discharge machining (EDM). The tensile sample geometry is presented in Figure 4-2. The gauge length and the cross-section area in the gauge zone were carefully measured by a digital micrometer (\pm 0.001 mm resolution) for all samples before tensile testing. Three tests were performed on each alloy to make ensure that the results were reproducible.



Figure 4-2: Sample geometry for all tensile tests (all dimensions are in mm and the sample thickness is the same as the sheet thickness).

The data obtained from uniaxial tensile testing until fracture was used to plot the true stress-strain curves and determine various tensile characteristics such as yield stress (YS), ultimate tensile strength (UTS) and uniform elongation. In addition to true stress-strain curves, differential true stress-strain plots were calculated to investigate the work hardening behaviour of the experimental alloys. The Considère criterion (Eq. 4-1) was

then used to determine the onset of tensile instability (necking), with σ and ϵ denoting true stress and true strain, respectively.

$$\frac{d\sigma}{d\varepsilon} = \sigma \tag{4-1}$$

The fracture surfaces were viewed by SEM to determine the fracture mode.

Interrupted tensile tests were carried out to study alloy microstructural evolution with tensile deformation and to further investigate the deformation modes of both the homogenous and carbon graded alloys via SEM + EBSD and XRD. In the case of the interrupted tensile samples, the tensile experiments were stopped at true strains of 0.02, 0.05, 0.1, 0.2, 0.3 and 0.45. Small pieces were cut from the gauge zone of these deformed samples and their cross sections prepared for EBSD and XRD analyses. EBSD observations were used to determine the deformation product type (mechanical twins or ε -martensites) and thus the critical strain for onset. XRD analyses were also employed to determine the volume fraction of ε -martensite within each sample. The details of the EBSD and XRD analyses are provided below.

4.5 Characterization Techniques

Different analytical techniques for materials characterization were utilized in this work to aid in determining the metallurgical mechanisms involved. The specific purpose of choosing each technique in addition to the analytical parameters involved will be described in the following sections.

4.5.1 Glow Discharge Optical Emission Spectroscopy (GDOES)

This technique is generally used for rapid, direct bulk chemical and compositional depth profiling analysis of different solids including metals, ceramics, glasses, polymers and powders. In the current work, GDOES was used via a GD Profiler from HORIBA Jobin Yvon to determine the chemical analysis for all elements other than carbon and sulfur for the as-received alloys. Calibration of this analytical method was carried out with a set of reference samples of compositions similar to the experimental samples. For carbon and sulfur analysis, the combustion method was employed. GDOES was also employed to obtain carbon concentration profiles of all carbon graded steels. Carbon concentration profiles were obtained by removing successive surface layers by grinding followed by carbon analysis, allowing C concentration profiles versus depth to be constructed. For the homogenous steels, the carbon concentration was also analyzed by GDOES at different surface layer depths to ensure that the carbon distribution was uniform through the thickness of the samples. However, the bulk carbon content of the homogeneous alloys was also obtained by the combustion method.

4.5.2 Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP – OES)

This method was used for quantitative measurement of the elemental compositions of the experimental materials. The only elements which cannot be analyzed by this method are C, O, H, N and the noble gases. In this work, a Vista-Pro ICP – OES from Varian was used to measure the chemical concentrations of all elements except carbon and sulfur in the as-received alloys. Standard solutions with known concentrations of the elements of interest were introduced into the machine and the intensity of the

characteristic emission for each element in the standard was measured. These intensities were plotted versus the standard concentrations to create a calibration curve. The applied power, plasma flow and nebulizer flow were 1.2 kW, 15 L/min and 0.7 L/min, respectively. The ICP – OES instrument was cleaned by a blank solution (10 vol. % H₂SO₄ solution) to remove contaminants in the tubes.

4.5.3 Carbon and Sulfur Combustion Analysis

A LECO HF-400 combustion analyzer was used in this research for the measurement of carbon and sulfur in the as-received and homogenous (as heat-treated) alloys. The instrument was capable of detecting carbon and sulfur down to approximately 0.0001 wt. % in a metal sample. Standards were used with each sample which had a carbon and sulfur content as close to the sample as possible.

4.5.4 Scanning Electron Microscopy (SEM) and Electron Backscatter Diffraction (EBSD)

Secondary electron imaging was used for fractographic examination of all experimental alloys in this work. An acceleration voltage of 10 keV and a working distance of 10 mm were applied throughout the study using a JEOL JSM-6610LV scanning electron microscope.

In the case of high manganese steels, deformation twins and ε -martensite platelets are microstructurally similar and cannot be distinguished via imaging techniques such as conventional SEM or optical microscopy. However, by using EBSD, it is possible to identify both twins and ε -martensite platelets due to orientation contrast.

All EBSD analyses in the current work were carried out using a JEOL JSM-7000F FEG-SEM equipped with a Charge-Coupled Device (CCD) detector. The acceleration voltage was set at 20 keV and a working distance of 18.4 mm was used for all analyses. The sample was tilted 70° towards the EBSD detector to increase the backscattered electron yield. The magnification was 400x in all cases. For all EBSD phase mappings, the step size was set at 0.4 µm to obtain sufficient resolution within a large enough map.

The "Flamenco" program in the HKL software package was used for the operation and control of all EBSD analyses. In order to distinguish between austenite (FCC) and ε martensite (HCP) phases, the maximum/minimum band detection was set at 5/8. The Mean Angular Deviation (MAD) limit was also decreased to 1.0° to obtain more accurate indexing results.

The "Tango" program in the HKL software package was used for post-analysis of the EBSD results to obtain phase maps. The EBSD phase maps assigned different colours to the various phases present within a map. Grain boundaries were delineated by considering misorientation angles larger than 15° whereas twin boundaries were determined by setting the twinning plane as {111} and the misorientation angle as 60° with a deviation of \pm 3°. A point counting method developed by Renard and Jacques (Renard and Jacques, 2012) was used to estimate the volume fraction of twins based on the EBSD phase maps.

4.5.5 X-Ray Diffraction (XRD)

X-ray diffraction analysis was carried out using a Bruker D8 Discover diffractometer to quantitatively determine the volume fraction of ε -martensite in samples. The X-ray beam was generated from a Co source with an average K α line of 1.7902% using a voltage and current of 35 kV and 45 mA, respectively. Spectra were taken in the range of 20 from 36° to 120° with a 0.03° step size for a dwell time of 1.3 s per step. Since sample cross-sections were analyzed in all cases, a 1.0 mm diameter aperture was selected for all analyses. The sample was moved back and forth along its length and also rotated continuously to mitigate any preferred orientation effects on the analysis. Three runs were made for each sample and the average of the results taken.

The 2D XRD data were collected using the Diffrac.Measurement Center (version 3) program. Moreover, the Diffrac.Pilot (version 2011.4) program was used to convert the 2D data into a conventional intensity vs. 2θ plot. Quantitative XRD phase analysis was performed using the TOPAS 4.2 software.

4.6 Fracture Analysis

The fracture behaviour of both the homogenous and carbon graded alloys was investigated. The fracture surfaces were examined using SEM to identify the sample fracture mode (s). Through-thickness cross sections of the fractured samples were also metallographically prepared and analyzed by SEM to observe deformation products and fracture characteristics such as the formation of cracks and voids.

5 EXPERIMENTAL RESULTS FOR FE-22MN-C ALLOYS

In this chapter, the experimental results for both the homogenous and carbon graded Fe-22Mn-C alloys are presented. Homogenous alloy results are presented in §5.1 followed by the carbon graded alloy results in §5.2. The experimental results for Fe-30Mn-C alloys will be presented in Chapter 6. A detailed discussion of all of the experimental results can be found in Chapter 7.

5.1 Homogenous Fe-22Mn-C Alloys

In this section, the carbon content and microstructure of the as heat-treated alloys will be presented, followed by results concerning the tensile behaviour and microstructural evolution during tensile deformation. Finally, a fracture analysis of all homogenous Fe-22Mn-C alloys will be presented.

5.1.1 As Heat-Treated Alloy Carbon Content and Microstructure

The as-received Fe-22Mn-0.6C steel was heat treated following the methods mentioned in Chapter 4 to obtain three homogenous steels with lower carbon contents. The carbon contents of the as heat-treated homogenous Fe-22Mn-C alloys as well as the stacking fault energy (SFE) values determined using available models can be found in Table 5-1. The measured carbon contents were very close to the target levels for all the alloys except the 0C alloy. According to the SFE model of Dumay et al. (Dumay et al., 2008), the deformation products were expected to be mechanical twins for the 0.4C and 0.6C alloys and mechanical ε -martensite for the 0C, 0.2C and 0.4C alloys. This model also predicted the presence of thermal ε -martensite in the as heat-treated microstructures

Ph.D. Thesis – M. Ghasri Khouzani McMast

McMaster – Materials Science & Engineering.

of the 0C and 0.2C alloys. The SFE model developed by Nakano and Jacques (Nakano and Jacques, 2010) also predicted mechanical ε-martensite formation for the 0C, 0.2C and 0.4C alloys without providing any comment on mechanical twinning. This model also predicted the presence of thermal ε-martensite in the microstructure of the as heat-treated 0C and 0.2C alloys. The SFE model proposed by Saeed-Akbari et al. (Saeed-Akbari et al., 2009), however, predicted mechanical ε-martensite formation for all homogenous alloys listed in Table 5-1. As was seen in the Nakano and Jacques model (Nakano and Jacques, 2010), this model does not contain a SFE range for mechanical twinning. Moreover, it should be noted that the results listed in Table 5-1 indicate that the Nakano and Jacques model (Nakano and Jacques, 2010) contains a weaker effect of the alloy carbon content on the SFE value compared to the other models.

Table 5-1. Carbon content and computed SFE values for all homogenous Fe-22Mn-C alloys.

Alloy Name	Carbon content (wt. %)	SFE (mJ/m ²) by (Dumay et al., 2008)	SFE (mJ/m ²) by (Nakano and Jacques, 2010)	SFE (mJ/m ²) by (Saeed-Akbari et al., 2009)
0C	0.06	-1.5	26.1	-4.9
0.2C	0.19	5.5	29.2	-0.6
0.4C	0.42	15.0	33.4	6.7
0.6C	0.57	19.7	36.8	11.4

The average grain size of the as heat-treated samples was $102 \pm 3 \mu m$, $107 \pm 4 \mu m$, $109 \pm 2 \mu m$ and $113 \pm 3 \mu m$ for the 0C, 0.2C, 0.4C and 0.6C alloys, respectively. From this data, it can be concluded that there were only minor differences in the grain sizes of the alloys. Since ε -martensite and twins are microstructurally similar to each

other and cannot be differentiated by conventional SEM, EBSD was used for the microstructural analysis of all alloys. For all EBSD phase maps presented, it should be noted that FCC austenite is denoted red, HCP ε-martensite blue, twin boundaries in yellow and un-indexed areas in black. Moreover, grain boundaries are highlighted as black lines.

Figure 5-1 illustrates typical EBSD phase maps from the cross-sections of the as heat-treated homogenous alloys. The 0C and 0.2C alloys comprised a dual-phase microstructure of equiaxed austenite and thermal ε -martensite, where the volume fraction of ε -martensite in the 0C alloy was significantly larger versus that of the 0.2C alloy (Figure 5-1(a) and (b)). By contrast, for the 0.4C and 0.6C alloys, the microstructures comprised an equiaxed austenitic matrix with some annealing twins and no significant ε -martensite (Figure 5-1(c) and (d)). This increase of the austenite volume fraction in the as heat-treated microstructures with increasing carbon content can be attributed to carbon generally increasing the SFE for a given alloy manganese level. For all homogenous alloys, the as heat-treated microstructures generally confirmed the predictions of the Dumay et al. (Dumay et al., 2008) and Nakano and Jacques (Nakano and Jacques, 2010) SFE models.



Figure 5-1: Typical EBSD phase maps from the cross-section of as heat-treated homogenous Fe-22Mn-C alloys.

5.1.2 Tensile Behaviour

In the present sub-section, the tensile behaviour of the homogenous Fe-22Mn-C alloys at room temperature will be presented. For each experimental alloy, a minimum of three uniaxial tensile tests were performed to assess the repeatability of the results. True stress-true strain curves of the as heat-treated 0C alloy at room temperature are presented in Figure 5-2. From this, it can be seen that the results of the three uniaxial tensile tests

were consistent. For later comparison and discussion, the results of test 2 were selected as being representative of the tensile behaviour for the 0C alloy. The tensile results for the other alloys were similarly repeatable, a complete compilation of which can be found in Appendix A. For all other alloys, a representative tensile curve was selected for further discussion.



Figure 5-2: Room temperature true stress-true strain curves for the homogenous Fe-22Mn-0C alloy.

Representative room temperature true stress-true strain curves for all homogenous Fe-22Mn-C alloys as a function of alloy C content are shown in Figure 5-3(a) and the Ph.D. Thesis – M. Ghasri Khouzani

McMaster – Materials Science & Engineering.

derived tensile properties for all alloys listed in Table 5-2. All tensile curves exhibited continuous yielding. For the 0.2C, 0.4C and 0.6C alloys, where the as heat-treated microstructures contained insignificant or minor amounts of thermal ε-martensite, the yield stress decreased with decreasing carbon content (Figure 5-3(a), Table 5-2). This can be explained by the solid solution hardening effect of carbon in austenite (Bouaziz et al., 2011b, Ohkubo et al., 1994). The 0C alloy did not obey this trend because its as heat-treated microstructure contained a significant fraction of thermal ε-martensite (see Figure 5-1(a)), which is harder than austenite and resulted in flow curves and work hardening histories for this alloy resembling those of a conventional low alloy dual phase steel.



(a) Representative true stress-true strain curves as a function of alloy C content



(b) Corresponding work hardening rate versus true stress as a function of alloy C content

Figure 5-3: Room temperature tensile response of homogenous Fe-22Mn-C alloys.

Alloy Name	Yield Stress (MPa)	UTS (MPa)	Uniform Elongation
0C	251 ± 4	791 ± 9	0.18 ± 0.02
0.2C	172 ± 3	999 ± 7	0.36 ± 0.03
0.4C	223 ± 4	1221 ± 8	0.52 ± 0.03
0.6C	265 ± 6	1372 ± 11	0.57 ± 0.05

Table 5-2. Room temperature tensile characteristics of homogenous Fe-22Mn-C alloys.

It can also be seen from Table 5-2 that the ultimate tensile strength (UTS) and uniform elongation increased with increasing alloy carbon content, which can be attributed to the sustained high work hardening rates exhibited by these alloys as well as the solid solution hardening effect of carbon (Figure 5-3(b)). The UTS value of the 0.6C alloy was lower while its uniform elongation was larger than previously reported for this steel (Allain et al., 2004b, Bouaziz et al., 2008, Barbier et al., 2009, Bouaziz et al., 2011a). This can be explained by the larger grain size (113 μ m) of this steel prior to tensile testing versus 15 µm (Allain et al., 2004b), 25 µm (Bouaziz et al., 2008) and 2.6 μm (Barbier et al., 2009). The flow curves of the 0.4C and 0.6C alloys showed significant serrations, attributed to the Portevin-LeChatelier (PLC) or dynamic strain aging (DSA) effect (Figure 5-3(a)) (Chen et al., 2007, Renard et al., 2010). Serrations were not observed in the flow curves of the 0C and 0.2C alloys. However, these alloys had a higher initial work hardening rate than the 0.4C and 0.6C alloys (Figure 5-3(b)), which rapidly declined with increasing strain. As shown by Liang et al. (Liang et al., 2009), these flow curves are characteristic of a high Mn steel in which the dominant deformation product was ε -martensite – i.e. a TRIP alloy.

Further inspection of Figure 5-3(b) shows that the experimental alloys exhibited distinctly different work hardening histories, where the 0C and 0.2C alloys showed relatively high work hardening (i.e. $d\sigma/d\epsilon$) values at low strains which declined rapidly with increasing strain. These work hardening histories are consistent with those observed by Yang et al. (Yang et al., 2010) and Koyama et al. (Koyama et al., 2011) for their alloys in which ε -martensite was the primary deformation product. However, the 0.4C and 0.6C alloys showed several distinct stages to their $d\sigma/d\epsilon$ values, where the $d\sigma/d\epsilon$ values for the 0.4C initially declined with increasing strain until $\sigma \approx 300$ MPa ($\varepsilon \approx 0.05$) before attaining a maximum at $\sigma \approx 810$ MPa ($\varepsilon \approx 0.29$), after which the work hardening rates declined to fracture (Figure 5-3(b)). Similarly, the $d\sigma/d\epsilon$ values for the 0.6C alloy initially declined with increasing strain until $\sigma \approx 400$ MPa ($\epsilon \approx 0.08$), after which they increased until $\sigma \approx$ 860 MPa ($\epsilon \approx 0.30$), followed by declining work hardening rates to fracture. This behaviour is consistent with stages A through C of the work hardening curves of a Fe-20Mn-1.2C TWIP steel as described by Renard and Jacques (Renard and Jacques, 2012) and is consistent with the work hardening behaviour generally observed for high-Mn TWIP steels (Chen et al., 2007, Barbier et al., 2009, Renard et al., 2010, Yang et al., 2010).

5.1.3 Microstructural Evolution with Tensile Deformation

To determine the products in the deformed microstructures, XRD and EBSD analyses were performed on all homogenous alloys as a function of plastic strain. A plot of ε -martensite volume fraction as a function of true strain for all homogenous Fe-22Mn-

C alloys is depicted in Figure 5-4. For the undeformed microstructures (at true strain of 0), the ε -martensite volume fraction was approximately zero for the 0.4 C and 0.6C steels, and was approximately 0.54 and 0.17 for the 0C and 0.2C alloys, respectively. This was consistent with the microstructural observation of as heat-treated alloys illustrated in Figure 5-1.



Figure 5-4: Evolution of ε -martensite volume fraction with tensile strain for all homogenous Fe-22Mn-C alloys, measured via XRD.

Further inspection of Figure 5-4 will show that the ε -martensite volume fraction increase in the 0C and 0.2C alloys was the most significant for 0.1 true strain although it continued to increase until failure. The saturated volume fraction of ε -martensite in the

microstructure was inversely proportional to the alloy C level and, by implication, the SFE value of the alloy. This will be discussed in detail in Chapter 7. It can also be seen that for these two alloys, the mechanical $\epsilon \gamma$ -martensite transformation followed a sigmoidal kinetic, with a significant portion of the transformation occurring between $\epsilon = 0$ and $\epsilon = 0.1$, and where the rate of ϵ -martensite transformation declined significantly for $\epsilon \ge 0.1$ and that, in the case of the 0.2C alloy, became saturated for $\epsilon \ge 0.3$.

EBSD phase maps from cross-sections of the deformed OC alloy are illustrated in Figure 5-5. It can be seen that the deformed microstructures were a mixture of austenite and ε -martensite where the amount of ε -martensite was significantly higher than that observed in the as heat-treated microstructure (Figure 5-1(a)) and increased with increasing tensile strain, indicating that the austenite matrix transformed to ε -martensite during tensile testing. These observations are in agreement with the XRD analysis shown in Figure 5-4. No deformation twins were detected in the EBSD phase maps, leading to the conclusion that the primary deformation mode for the 0C alloy was the TRIP effect – i.e. dislocation glide and ε -martensite formation. This explains the high initial work hardening rate and relatively poor uniform elongation observed in Figure 5-3.





Figure 5-5: EBSD phase maps of cross-sections for 0C alloy as a function of true strain.

Figure 5-6 illustrates EBSD phase maps from the cross-sections of the deformed 0.2C alloy. At a true strain (ε) of 0.02 (Figure 5-6(a)), similar to the as heat-treated steel (Figure 5-1(b)), the microstructure comprised an austenite matrix, thermal ε -martensite and some annealing twins. At $\varepsilon = 0.05$ (Figure 5-6(b)), the austenite matrix started to transform to ε -martensite platelets. For the 0.2C alloy deformed to $\varepsilon = 0.1$ ((Figure 5-6(c)), a significant increase in the amount of ε -martensite was observed, with large platelets intersecting other ε -martensite plates. At higher levels of deformation (Figure

5-6(d)-(f)), large portions of austenite transformed to ε -martensite. A few annealing twins were observed within the various maps and the twin population was not seen to increase as a function of tensile strain. The EBSD phase maps correlated with the XRD data (Figure 5-4), where the ε -martensite volume fraction increased with true strain with the most significant increase being observed for $\varepsilon = 0.1$. No significant α' martensite was detected by EBSD or XRD. From both the EBSD phase maps and the XRD data, the primary deformation product for the 0.2C alloy was ε -martensite, resulting in a TRIP effect. This explains the high initial work hardening rate and relatively low uniform elongation observed in Figure 5-3. These results were consistent with the predictions of several SFE models (Dumay et al., 2008, Nakano and Jacques, 2010, Saeed-Akbari et al., 2009).





(a) ε=0.02





50 µm



(d) ε=0.2



Figure 5-6: EBSD phase maps of cross-sections for 0.2C alloy as a function of true strain.

EBSD phase maps from the cross-sections of the deformed 0.4C alloy are illustrated in Figure 5-7. At low strain levels (Figure 5-7(a) and (b)) the microstructure comprised an austenitic matrix with some annealing twins. At higher strain levels (Figure 5-7(c), (d), (e) and (f)), the deformation products were mechanical twins with a small volume fraction of ε -martensite being observed. This latter observation was consistent with the XRD data in Figure 5-4, which showed that there was a small but significant volume fraction of mechanical ε -martensite in the microstructure for $\varepsilon \ge 0.1$. From Figure 5-4, it can be seen that the majority of the ε -martensite present in the deformed microstructure evolved between applied strains of 0.05 and 0.1, after which the microstructure appears to have been saturated with this phase. This significant increase in ε-martensite volume fraction was also observed for the 0C and 0.2C alloys. This indicates that the driving force for $\gamma \rightarrow \varepsilon$ martensite transformation decreased at higher strain levels. It is worth noting that EBSD detected a lower increase in the amount of ε -martensite from ε =0.05 to ε =0.1, compared to that detected by XRD. From the EBSD analysis alone, the maps could not quantify the amounts of austenite and ε -martensite as the chosen mapping area may not have been representative of the whole sample, as would be the case for XRD.





(a) ε=0.02



(b) ε=0.05



(c) ε=0.1





Figure 5-7: EBSD phase maps of cross-sections for 0.4C alloy as a function of true strain.

A point counting method developed by Renard and Jacques (Renard and Jacques, 2012) was used to estimate the volume fraction of twins based on the EBSD phase maps. Figure 5-8 depicts the evolution of twin volume fraction with true strain for the 0.4C and 0.6C alloys. In the case of 0.4C alloy, the volume fraction of twins increased slightly up to $\varepsilon = 0.1$. Further deformation led to a sharp increase of twin volume fraction before the curve saturated. It should also be noted that the saturated twin density was relatively low versus that of the 0.6C alloy. Further examination of Figure 5-7 reveals that the twin population for different grains was not the same, which is consistent with general findings in the literature concerning the effect of the local Schmid factor on twin activation (Allain et al., 2002, Barbier et al., 2009, Gutierrez-Urrutia et al., 2010, Yang et al., 2006).

From both the EBSD phase maps (Figure 5-7) and XRD data (Figure 5-4), a combination of TWIP and TRIP effects – i.e. dislocation glide, mechanical twinning and mechanical ε -martensite formation – was found to be the deformation mode for the 0.4C alloy. This is similar to deformation products reported for a Fe-22Mn-0.376C steel when deformation was made through cold rolling (Lü et al., 2010). The experimental results for the 0.4C alloy also confirmed the predictions of the SFE model proposed by Dumay et al. (Dumay et al., 2008) (see Table 5-1).



Figure 5-8: Twin volume fraction as a function of strain for the 0.4C and 0.6C alloys.

Figure 5-9 illustrates EBSD phase maps from the cross-sections of the deformed 0.6C alloy. For low strain levels (Figure 5-9(a) and (b)), the microstructure consisted of a fully austenitic matrix with some annealing twins, which was similar to the as heat-treated microstructure (Figure 5-1(d)). As the true strain approached 0.1 (Figure 5-9(c)), mechanical twins were first observed, and the twin volume fraction sigmoidally increased with increasing deformation until necking with a trend similar to that observed for the 0.4C alloy (Figure 5-8). It should also be noted, however, that the twin volume fraction at higher strains was significantly higher versus that of the 0.4C alloy. This trend was also similar to the ε -martensite volume fraction change with true strain, where a sigmoidal

Ph.D. Thesis – M. Ghasri KhouzaniMcMaster – Materials Science & Engineering.characteristic was observed. However, the true strains for the activation of ε -martensiteand twins were not the same (see Figure 5-4 and Figure 5-8).

It should be noted that the onset of twin formation coincided with the observed increase in the work hardening rate for this alloy (Figure 5-3(b)), implying that the socalled "dynamic Hall-Petch effect" of twins refining the microstructure and, thereby, reducing the dislocation mean free path, was a primary determinant of the observed increase in $d\sigma/d\epsilon$ (Allain et al., 2004a, Bouaziz and Guelton, 2001, Shiekhelsouk et al., 2009). Further inspection of Figure 5-9 reveals that the population of mechanical twins for different grains was not the same, which is in agreement with the general findings in literature concerning the effect of grain orientation on the stress required for twin activation (Allain et al., 2002, Barbier et al., 2009, Gutierrez-Urrutia et al., 2010, Yang et al., 2006). No ε -martensite was observed in the microstructure of this alloy for all applied strains, as shown in Figure 5-9. The XRD data for this alloy confirmed this observation, where no significant ε -martensite was detected for strains up to failure (see Figure 5-4). Thus, it can be concluded that dislocation glide and mechanical twinning (i.e. the TWIP effect) was the dominant deformation mode for this alloy. This is consistent with the previous work on the deformation mode of Fe-22Mn-0.6C steel (Allain et al., 2004a, Allain et al., 2004b, Allain et al., 2004c).



(a) ε=0.02



(b) ε=0.05



(c) ε=0.1

(d) ε=0.2



Figure 5-9: EBSD phase maps of cross-sections for 0.6C alloy as a function of true strain.

5.1.4 Fractography

In order to observe features presented in the damage zone and identify the main fracture mode for all homogenous alloys, the fracture surfaces and sample cross-sections near the fracture surfaces were examined after uniaxial tensile testing to failure. Figure 5-10 illustrates the SEM micrographs from the fracture surface ((a) and (b)) and the crosssection near the fracture surface (c), sectioned along the tensile direction, for the Fe-22Mn-0C alloy. The fracture surface displayed micro-voids, micro-cracks, a large area fraction of cleavage facets and some ductile tearing features (Figure 5-10(a) and (b)). This is not surprising considering the large volume fraction of the relatively brittle ε -martensite phase detected by XRD and EBSD (Figure 5-4 and Figure 5-5). In the cross-section of the fracture sample, micro-voids and cracks were frequently observed along matrix/inclusions interfaces, as illustrated in Figure 5-10(c), indicating that these were significant sites for damage nucleation and void growth. This finding is somewhat at odds with the cleavage dominated fracture surfaces shown in Figure 5-10(a) and (b), which indicate that void nucleation at the austenite/ɛ-martensite interface played a significant role in the damage process. This also disagrees with the findings of Liang (Liang, 2008), Yang (Yang, 2010), Koyama et al. (Koyama et al., 2011) and Lee et al. (Lee et al., 2014), who determined that void nucleation at the austenite/ ε -martensite interface was the dominant damage mechanism for their *ɛ*-martensite formation dominated alloys tensile tested to failure.



(a) low magnification view

(b) higher magnification view



(c) void nucleation at the matrix/inclusions interface

Figure 5-10: SEM fractographs of homogenous Fe-22Mn-0C alloy.

Figure 5-11 displays the SEM micrographs from the fracture surface ((a) and (b)) and the cross-section near the fracture surface (c), sectioned along the tensile direction, for the Fe-22Mn-0.2C steel. Various fracture features observed within the fracture surface included micro-cracks, micro-voids, a large area fraction of cleavage facets and some ductile tearing features (Figure 5-11(a) and (b)). The 0.2C alloy showed a lower area fraction of cleavage facets and more ductile tearing features versus the 0C alloy. This difference can be explained by considering the lesser volume fraction of ε -martensite within this alloy at high strains as determined by XRD and EBSD techniques (Figure 5-4
and Figure 5-6). In the cross-section of the fracture sample, micro-voids and cracks were observed along matrix/inclusions interfaces, as displayed in Figure 5-11(c). Thus, the dominant damage mechanism for the 0.2C alloy was found to be void nucleation and growth at the matrix/inclusions interfaces. This finding is not consistent with the cleavage-like fracture surfaces shown in Figure 5-11(a) and (b), indicating that void nucleation at the austenite/ ϵ -martensite comprised a major contribution in the damage process. This is also against the general finding in the literature, which determined void nucleation at the austenite/ ϵ -martensite interface as the dominant damage mechanism for their ϵ -martensite formation dominated alloys (Koyama et al., 2011, Lee et al., 2014, Liang, 2008, Yang, 2010).



(a) low magnification view

(b) higher magnification view



(c) void nucleation at the matrix/inclusions interface

Figure 5-11: SEM fractographs of homogenous Fe-22Mn-0.2C alloy.

Figure 5-12 illustrates the SEM micrographs from the fracture surface ((a) and (b)) and the cross-section near the fracture surface (c), sectioned along the tensile direction, for the Fe-22Mn-0.4C steel. The fracture features mainly comprised micro-voids and ductile tearing features (Figure 5-12(a) and (b)). However, some cleavage facets and micro-cracks were also observed. The relative population of these features is in agreement with the small volume fractions of ε -martensite detected by XRD and EBSD analyses in the fractured sample microstructures, as shown in Figure 5-4 and Figure 5-7, respectively. In the cross-section of the alloy near the fracture surface (Figure 5-12(c)),

significant occurrences of grain boundary separation and void nucleation at inclusions were observed, indicating that these were the dominant damage modes for this alloy.



(a) low magnification view

(b) higher magnification view



(c) crack at a grain boundary

Figure 5-12: SEM fractographs of homogenous Fe-22Mn-0.4C alloy.

Figure 5-13 displays the SEM micrographs from the fracture surface ((a) and (b)) and the cross-section near fracture surface (c), sectioned along the tensile direction, for the Fe-22Mn-0.6C steel. In the fracture surface (Figure 5-13(a) and (b)), micro-voids and ductile tearing features were observed with no significant cleavage facets being observed. In the fracture cross-sections of the alloy (Figure 5-13(c)), decohesion of a series of

inclusions along a grain boundary was observed in addition to void nucleation at grain boundaries. Thus, the damage mode for this alloy was concluded to be void nucleation at grain boundaries and inclusion/matrix interfaces.



(a) low magnification view

(b) higher magnification view



(c) decohesion of inclusions

Figure 5-13: SEM fractographs of homogenous Fe-22Mn-0.6C alloy.

5.1.5 Summary of Homogenous Fe-22Mn-C Alloys

The as heat-treated 0C and 0.2C alloys had a dual phase microstructure of austenite and thermal ε -martensite, whereas the 0.4C and 0.6C alloys had a fully austenitic microstructure prior to deformation. The deformation products for the 0C and

Ph.D. Thesis – M. Ghasri Khouzani

McMaster – Materials Science & Engineering.

0.2C alloys were found to be mechanical ε -martensite. This led to high initial work hardening rate but low uniform elongation for the 0C and 0.2C alloys versus 0.4C and 0.6C alloys. Mechanical twins were found to be the dominant deformation products for the 0.6C alloy, which resulted in this alloy displaying high sustained work hardening rates. For this reason, this alloy showed the best mechanical properties among the homogenous Fe-22Mn-C alloys. The deformation products for the 0.4C alloy were a mixture of mechanical twins and mechanical ε -martensite. This alloy displayed a work hardening rate evolution typical of a mechanical twin formation steel, but with lower sustained high work hardening rates versus the 0.6C alloy.

It was determined that both the ε -martensite and twin formation followed a sigmoidal kinetic with strain. In the case of the 0C, 0.2C and 0.4C alloys – all of which had significant ε -martensite contents – the saturation volume fraction of ε -martensite was inversely proportional to the alloy C content and, by inference, the alloy SFE. For the twin forming 0.4C and 0.6C alloys, it was found that the saturation volume fraction of twins was directly proportional to the alloy C contents and, by implication, the alloy SFE.

5.2 Carbon Graded Fe-22Mn-C Alloys

In this section, the experimental results for carbon graded Fe-22Mn-C alloys will be presented. Carbon concentration profiles and the microstructures of the as decarburized alloys will be presented first, followed by results concerning the tensile behaviour and microstructural evolution during tensile deformation. Finally, a fracture analysis of the carbon graded Fe-22Mn-C alloys will be presented.

5.2.1 As Decarburized Alloy Carbon Concentration Profiles and SFE

As-received Fe-22Mn-0.6C steels were decarburized following the methods mentioned in Chapter 4 to obtain three steels with varying carbon gradients. The decarburization parameters are summarized in Table 5-3.

Table 5-3. Decarburization conditions for carbon graded Fe-22Mn-C alloys.

Alloy Name	Decarburization Atmosphere	Decarburization Time
G1	CO ₂ (99.9 %)	4 h
G2	wet H ₂ (5.2 %)+Ar (94.8 %)	1 h
G3	wet H ₂ (5.2 %)+Ar (94.8 %)	3 h

Carbon concentration profiles from the surface to the core for all the graded alloys are depicted in Figure 5-14. For all alloys, the carbon concentration profiles were assumed to be symmetric across the centre plane of the samples. This assumption was verified by microstructural observations, where the observed edge was not necessarily the same as the one already used for carbon concentration measurements. For the G1 steel, the carbon concentration at the surface was higher than that of the G2 and G3 steels,

consistent with CO_2 being a weaker decarburizing agent than wet hydrogen. It can also be seen that the carbon concentration gradient of the G2 steel was steeper than the G1 and G3 steels because its decarburization time was shorter (1 h). In the case of the G3 alloy, it should be noted that the carbon content at the core was significantly lower (approximately 0.35 wt. %) than that observed for the G1 and G2 alloys, consistent with the longer decarburization time (3 h) employed for this alloy.



Figure 5-14: Carbon concentration profile for the carbon graded Fe-22Mn-C steels. Note that the total sheet thickness was 1.5 mm or 1500 microns and that symmetry was assumed.

Figure 5-15 presents the manganese concentration profiles for the as-decarburized carbon graded Fe-22Mn-C alloys. For all alloys, the manganese concentration at the near

surface layer was lower versus the rest of the alloy. This can be attributed to the selective oxidation of manganese during decarburization. For deeper layers, the manganese concentration did not change significantly with the sample depth, being approximately 22 wt. %. This is in agreement with the bulk manganese content ($21.76 \pm .43$ wt. %) of the as-received Fe-22Mn-0.6C steel presented in Table 4-1.



Figure 5-15: Manganese concentration profile for the carbon graded Fe-22Mn-C alloys.

Using the carbon concentration profiles presented in Figure 5-14, the SFE values as a function of sample depth were calculated based on the Dumay et al. (Dumay et al., 2008) model for all the carbon graded Fe-22Mn-C steels. The resultant plots are depicted in Figure 5-16. For all carbon graded steels and the resultant SFE gradient, the as-

decarburized microstructure near the outer surface was expected to contain thermal ε martensite, with its volume fraction decreasing towards the core (Schumann, 1972). In the case of the G1 and G2 alloys, it was further expected that the deformed microstructures would exhibit mechanical ε -martensite deformation products at the outer surface and would mechanically twin at the core whereas the G3 alloy would form mechanical ε martensite at the outer surface and would have mixed ε -martensite/twin deformation products at the core (Dumay et al., 2008).



Figure 5-16: SFE versus sample depth for all carbon graded Fe-22Mn-C steels based on the Dumay et al. SFE model (Dumay et al., 2008).

5.2.2 As Heat-Treated Microstructures

EBSD was used for the microstructural analysis of all alloys. For all EBSD phase maps presented, FCC austenite is denoted in red, HCP ε -martensite blue, FCC manganese oxide (MnO) green, BCT α '-martensite white and un-indexed areas black. Grain boundaries are highlighted as black lines and twin boundaries in yellow.

Figure 5-17 illustrates the EBSD phase maps from the cross-sections of the as decarburized carbon graded Fe-22Mn-C steels. For all steels, a continuous external MnO layer was present, arising from the selective oxidation of manganese during the decarburization process. This layer was partially removed from the G2 steel surface during metallographic preparation. There was also a thermal α '-martensite layer observed immediately below the MnO external film, in which some MnO particles were observed. The manganese content of the α '-martensite layer was measured by EDS spot analysis to be approximately 4 wt. %. This indicates that the α '-martensite formed due to manganese depletion of the surface layer. Meguerian (Meguerian, 2007) has already reported formation of thermal α '-martensite for this manganese level at similar C levels.



McMaster – Materials Science & Engineering.



(c) G3 (0-250 μ m from surface)

(d) G3 (250-500 µm from surface)

Figure 5-17: EBSD phase maps from cross-section of as decarburized carbon graded Fe-22Mn-C steels.

Further examination of Figure 5-17 shows that for all the as decarburized carbon graded steels, the microstructure near the outer surface comprised a dual phase layer of austenite and thermal ɛ-martensite, where the ɛ-martensite volume fraction decreased towards the core. It is interesting to note that the thermal ε -martensite laths stop halfway through some grains and at a reasonably uniform depth for each sample. This depth can be estimated to be $\approx 180 \,\mu\text{m}$ for the G1 and G2 alloys. Using the plots presented in Figure

5-16, the corresponding SFE value can be found to be $\approx 9 \text{ mJ/m}^2$. This SFE value can be taken as threshold for the formation of thermal ε-martensite. A similar SFE value was reported by Allain et al. (Allain et al., 2004b) as threshold for the thermal ε -martensite formation, when they superimposed the Schumann phase boundary (Schumann, 1972) for this transformation onto their SFE map. However, it should be noted that the Schumann boundary line for the thermal ε -martensite formation was based on chemical composition and not the SFE value. The dual phase layer for the G3 steel was thicker ($\approx 260 \ \mu m$) than that of the G1 and G2 alloys ($\approx 125 \ \mu m$) (Figure 5-17). This was not surprising considering the difference in SFE profiles for the graded steels (Figure 5-16), where the SFE was lower than the threshold value ($\approx 9 \text{ mJ/m}^2$) for more of the cross-section in the case of the G3 alloy (390 μ m). The discrepancy between the observed thickness of the dual-phase layer for the G3 alloy ($\approx 260 \,\mu\text{m}$) and the estimated one (390 μm) indicates that 9 mJ/m^2 can be considered as an indicative value of a hard rule. The core microstructure for all of the graded alloys comprised austenite with some annealing twins, of which the mid-thickness section of the microstructure seen in Figure 5-17(d) was typical.

5.2.3 Tensile Behaviour

In the present sub-section, the tensile behaviour of the carbon graded Fe-22Mn-C alloys at room temperature will be presented. For each experimental alloy, a minimum of three uniaxial tensile tests were performed to assess the consistency of the results. The tensile results for all the alloys were repeatable, a complete compilation of which can be found in Appendix A.

Typical room temperature true stress-true strain (σ - ϵ) plots for all the carbon graded Fe-22Mn-C steels are presented in Figure 5-18(a) and their tensile properties summarized in Table 5-4. All tensile curves exhibited continuous yielding. The G3 alloy had a lower yield stress relative to the G1 and G2 materials. This can be explained by the solid solution hardening effect of carbon in austenite (Bouaziz et al., 2011b, Ohkubo et al., 1994) when considering the lower overall carbon content of the as decarburized G3 alloy compared to the other two alloys (see Figure 5-14).

The flow curves of the G1 and G2 alloys showed significant serrations, attributed to the Portevin-LeChatelier (PLC) or dynamic strain aging (DSA) effect (Figure 5-18(a)) (Chen et al., 2007, Renard et al., 2010). This indicates that the G1 and G2 alloys had sufficient carbon at their core to promote the PLC effect. For the G3 alloy, however, the flow curve did not show any serrations (Figure 5-18(a)). This alloy had a higher initial work hardening rate than the G1 and G2 alloys (Figure 5-18(b)), which rapidly declined with increasing stress. As indicated by Liang et al. (Liang et al., 2009), Yang et al. (Yang et al., 2010) and Koyama et al. (Koyama et al., 2011), this flow curve is characteristic of a high manganese steel in which the dominant deformation product was ε -martensite – i.e. a TRIP alloy.



(b) Corresponding work hardening rate as a function of true stress

Figure 5-18: Tensile response of carbon graded Fe-22Mn-C alloys at room temperature.

Table 5-4. Room temperature tensile characteristics of the carbon graded Fe-22Mn-C alloys.

Alloy Name	Yield Stress (MPa)	UTS (MPa)	Uniform Elongation
G1	219 ± 4	936 ± 7	0.38 ± 0.04
G2	239 ± 5	1022 ± 7	0.38 ± 0.03
G3	206 ± 3	843 ± 9	0.26 ± 0.02

Further inspection of Figure 5-18(b) will show that the carbon graded alloys exhibited distinctly different work hardening histories, where the G3 alloy showed relatively high work hardening values at low strains which declined relatively rapidly with increasing strain. These work hardening histories are consistent with those observed in Figure 5-3(b) for the ε -martensite formation 0C and 0.2C alloys. Similar work hardening behaviours were also observed by Yang et al. (Yang et al., 2010) and Koyama et al. (Koyama et al., 2011) for their alloys in which ε -martensite was the primary deformation product. However, the G1 and G2 alloys showed several distinct stages to their $d\sigma/d\epsilon$ values, where the $d\sigma/d\epsilon$ values for the G1 initially declined with increasing strain until $\sigma \approx 350$ MPa ($\epsilon \approx 0.06$) before attaining a maximum at $\sigma \approx 765$ MPa ($\epsilon \approx$ 0.28), after which the work hardening rates declined to fracture (Figure 5-18(b)). Similarly, the $d\sigma/d\epsilon$ values for the G2 alloy initially declined with increasing strain until σ ≈ 400 MPa ($\epsilon \approx 0.08$), after which they increased until $\sigma \approx 780$ MPa ($\epsilon \approx 0.25$), followed by declining work hardening rates to fracture. This behaviour is consistent with the work hardening curves of the 0.4C and 0.6C alloys as shown in Figure 5-3(b) and is consistent with the work hardening behaviour generally observed for high-Mn TWIP steels (Chen et al., 2007, Barbier et al., 2009, Renard and Jacques, 2012, Renard et al., 2010, Yang et al., 2010).

5.2.4 Microstructural Evolution with Tensile Deformation

To determine the deformation products in the deformed microstructures, EBSD analyses were performed on all the carbon graded steels as a function of plastic strain. XRD analyses were also attempted in order to evaluate the microstructural evolution with tensile strain; however, the results were not useful due to apparatus limitations. The microstructural evolution results for the G1 alloy were similar to those of the G2 alloy and are not presented here. Full documentation of the microstructural evolution for the G1 alloy can be found in Appendix B. Figure 5-19 illustrates the EBSD phase maps from the near-surface cross-section (0-250 microns from the surface) of the G2 alloy as a function of strain. The external MnO film cannot be seen because it was removed during tensile deformation and metallographic preparation. For all applied strains, the near-surface microstructure of the G2 steel comprised austenite and *\varepsilon*-martensite, where the volume fraction of ε -martensite increased significantly with tensile strain versus the asdecarburized microstructure shown in Figure 5-17(b). This indicates that these mechanical martensitic transformation occurred during deformation of this near-surface layer of the cross-section. Although a few mechanical twins were occasionally observed within the near-surface deformed microstructures, their population was very low. Thus, the dominant deformation product for the surface layer (0-250 microns from the surface) of the G1 and G2 alloys was concluded to be mechanical *\varepsilon*-martensite.





Figure 5-19: EBSD phase maps from the near surface cross-section (0-250 microns from the surface) of the G2 alloy as a function of true strain.

The volume fractions of the ɛ-martensite were inferred from the EBSD phase maps using the HKL software. Plots of ε -martensite volume fraction as a function of applied strain for the G2 and G3 alloys are presented in Figure 5-20. The high fraction of un-indexed areas at high strains ($\varepsilon > 0.2$), was proportionally shared between austenite and ɛ-martensite. For this reason, the symbols of ɛ-martensite volume fractions at high strains ($\varepsilon > 0.2$) are hollow in Figure 5-20. For the near-surface layer of the G2 alloy, it

can be seen that the ε -martensite volume fraction increased with true strain until $\varepsilon = 0.2$. As was mentioned above, the increase in ε -martensite volume fraction was a consequence of the strain or stress induced $\gamma \rightarrow \varepsilon$ martensitic transformation. At higher strains ($\varepsilon > 0.2$), the formation of mechanical ε -martensite is likely to saturate, as it was observed for homogenous alloys (Figure 5-4). The sigmoidal-shape plot of the ε -martensite volume fraction evolution with true strain for the near-surface layer of the G2 alloy (Figure 5-20) was consistent with that was observed for the homogenous 0C and 0.2C alloys in Figure 5-4.



Figure 5-20: Evolution of ε -martensite volume fraction with applied strain for G2 and G3 alloys; data was obtained from EBSD phase maps using the HKL software.

The EBSD phase maps from the mid-thickness cross-section (250-500 microns from the surface) of the G2 alloy as a function of applied strain are illustrated in Figure 5-21. For all applied strains, the microstructure of the cross-section mid-thickness layer was composed of austenite and ε -martensite as well as deformation twins, where the population of mechanical twins and volume fraction of *ɛ*-martensite increased with increasing the tensile strain. Compared to phase maps observed for the outer layer of the G2 alloy (Figure 5-19), here the microstructure contained a higher population of mechanical twins and lower amount of mechanical *ɛ*-martensite. The latter is quantitatively shown in Figure 5-20, where the saturated volume fraction of ε -martensite decreased from 0.63 for the near-surface layer to 0.07 for mid-thickness layer. The difference in volume fraction of deformation products for these two layers of crosssection can be explained by considering the SFE profile for the G2 alloy (Figure 5-16), where the SFE increased to $10.5-15.1 \text{ mJ/m}^2$ for the mid-thickness. According to the microstructural evolution with tensile deformation presented in Figure 5-21, it can be concluded that the deformation product for this cross-section layer of G2 alloy was a mixture of mechanical twins and mechanical *ɛ*-martensite. The same deformation products were observed for the present homogenous 0.4C alloy (Figure 5-7) and also a homogenous Fe-22Mn-0.376C steel deformed through cold rolling (Lü et al., 2010).





Figure 5-21: EBSD phase maps from the mid-thickness cross-section (250-500 microns from the surface) of the G2 alloy as a function of true strain.

Figure 5-22 shows the evolution of the twin volume fraction with true strain based on the point counting method. For the mid-thickness layer of the G2 alloy, the volume fraction of twins increased slightly up to a.1. Further deformation led to a sharp increase of twin volume fraction until necking. This explained the increase in work hardening rate of G2 alloy, which was sustained to high strains (see Figure 5-18(b)).



Figure 5-22: Twin volume fraction as a function of strain for the G2 and G3 alloys.

Figure 5-23 illustrates the EBSD phase maps from the core cross-section (500-750 microns from the surface) of the G2 alloy as a function of applied strain. At $\varepsilon = 0.05$ (Figure 5-23(a)), the core microstructure of the G2 alloy comprised an equiaxed austenite matrix with some annealing twins. When the applied strain approached 0.1, mechanical twins started to appear within the core microstructure, as displayed in Figure 5-23(b). At high applied strains (Figure 5-23(c) and (d)), the mechanical twin population increased significantly with applied strain. However, the twin population for different grains was not the same, which is consistent with the general findings in the literature concerning the effect of the local Schmid factor on twin activation (Allain et al., 2002, Barbier et al., 2009, Gutierrez-Urrutia et al., 2010, Yang et al., 2006). From Figure 5-20, it can be found

that the volume fraction of ε -martensite in the core region was approximately zero for all applied strains, which was much lower than that of the outer layers of cross-section of this steel. This was attributed to the SFE profile of this steel (Figure 5-16), where SFE increased to 15.1-17.9 mJ/m² for the core layer. A higher volume fraction of mechanical twins was developed within the core microstructure compared to the mid-thickness layer, as shown in Figure 5-22. However, the trend of twin volume fraction with strain for core layer was similar to that of the mid-thickness layer with a slight increase up to ≈ 0.1 followed by a sharp increase until necking. Based on the EBSD phase maps observed in Figure 5-23, the dominant deformation product for this layer (500-750 microns from surface) of G2 steel was determined to be mechanical twins.



Ph.D. Thesis – M. Ghasri Khouzani

50 µm

(c) ε=0.2

McMaster – Materials Science & Engineering.

(d) ε=0.3

Figure 5-23: EBSD phase maps from the core cross-section (500-750 microns from the surface) of G2 alloy as a function of true strain.

50 µm

The EBSD phase maps from the near-surface cross-section (0-250 microns from the surface) of the G3 alloy as a function of tensile strain are displayed in Figure 5-24. The external MnO film cannot be seen because it was removed during tensile deformation and metallographic preparation. At $\varepsilon = 0.05$ (Figure 5-24(a)), the near-surface microstructure of the G3 alloy contained an austenite matrix, thermal ε -martensite and annealing twins, which was similar to the as-decarburized microstructure (Figure

5-17(c)). At ε =0.1 (Figure 5-24(b)), a significant increase in the amount of ε -martensite was observed, with large platelets intersecting other ε -martensite plates. At higher deformation levels (Figure 5-24(c) and (d)), large portions of austenite were transformed to mechanical ε -martensite with entire grains of ε -martensite. A few annealing twins were occasionally observed within the near-surface microstructure of the G3 alloy, where their population were not seen to increase as a function of strain (Figure 5-24). Thus, the dominant deformation product for the near-surface cross-section layer of the G3 alloy was concluded to be mechanical ε -martensite.

From Figure 5-20, it can be seen that for near-surface cross-section layer of the G3 alloy, the volume fraction of ε -martensite increased with increasing strain level until fracture. As it was mentioned earlier, the increase in volume fraction of ε -martensite was a result of the strain or stress induced $\gamma \rightarrow \varepsilon$ mechanical martensitic transformation. Based on observations for the homogenous alloys (Figure 5-4), the formation of mechanical ε -martensite is expected to decelerate at high strains ($\varepsilon > 0.2$). In this way, the initial high work hardening rate of this alloy, which declined at high strains, can be explained (Figure 5-18).





Figure 5-24: EBSD phase maps from the near surface cross-section (0-250 microns from the surface) of G3 alloy as a function of true strain.

Figure 5-25 illustrates the EBSD phase maps from the mid-thickness cross-section (250-500 microns from the surface) of the G3 alloy as a function of tensile strain. For all applied strains, the microstructure of the cross-section mid-thickness layer comprised an austenite matrix and ε -martensite as well as mechanical twins, where the volume fraction of ε -martensite and population of mechanical twins increased with increasing strain (Figure 5-20 and Figure 5-22). This indicates that strain induced $\varepsilon\gamma$ mechanical

martensitic transformation occurred during deformation. Some mechanical twins can also be observed in the deformed microstructures; however, their volume fraction was not high, as it can be seen in Figure 5-22. Compared to the phase maps presented for the nearsurface layer (0-250 microns from the surface) (Figure 5-24), the microstructures here contained a lower amount of ε -martensite and a higher population of mechanical twins. From Figure 5-20, it can be seen that the saturated volume fraction of ε -martensite decreased from 0.52 for the near-surface layer to 0.22 for the mid-thickness layer. The difference in volume fraction of deformation products for these two layers of the crosssection can be explained by considering the SFE profile for the G3 alloy (Figure 5-16), where the SFE increased to 6.8-10.3 mJ/m² for the mid-thickness. From Figure 5-22, it can be found that the volume fraction of mechanical twins for the mid-thickness layer of the G3 alloy was lower than that of the G2 alloy. Based on the EBSD phase maps shown in Figure 5-25, the dominant deformation product for this layer of the G3 steel was determined to be a mixture of mechanical ε -martensite and twins.





(c) ε=0.2

50 µm

The EBSD phase maps from the core cross-section (500-750 microns from the surface) of the G3 alloy as a function of applied strain are illustrated in Figure 5-26. At ε = 0.05 (Figure 5-26(a)), the cross-section mid-thickness microstructure was composed of an austenite matrix, mechanical ε -martensite and annealing twins; however, the volume fraction of the mechanical ε -martensite was not significant. As 0.1 true strain was approached (Figure 5-26(b)), the initiation of mechanical twins was observed as well as

the increase in the volume fraction of mechanical *ɛ*-martensite. Approaching a true strain of 0.2, Figure 5-26(c) displays the austenite matrix with more mechanical twinning and ε martensite formation throughout the grains. From these phase maps, the dominant deformation products for the cross-section core of the G3 alloy were found to be a combination of mechanical twins and *ɛ*-martensite. However, compared to the outer layers of the G3 alloy (Figure 5-24 and Figure 5-25), the microstructure contained a relatively small amount of mechanical *ɛ*-martensite and a higher population of mechanical twins, as highlighted in Figure 5-20 and Figure 5-22. From Figure 5-20, it can be seen the saturated volume fraction of ε -martensite decreased from 0.52 for the near-surface layer to 0.03 for the core layer. Moreover, from Figure 5-22, it can be found that the twin volume fraction of the core layer was higher than that of the mid-thickness layer for all applied strains. The difference in volume fraction of deformation products can be explained by considering the SFE profile of this alloy (Figure 5-16), where the SFE increased from the surface towards the core, reaching $10.3-12.7 \text{ mJ/m}^2$ for the core layer. Mixed deformation products were already observed for the homogenous 0.4C alloy in Figure 5-7, although the twin volume fraction was higher (Figure 5-4) and the ε martensite volume fraction was lower (see Figure 5-8) compared to the core layer of the G3 alloy. The latter can also be attributed to the higher SFE (15.0 mJ/m²) of the 0.4C alloy.









Figure 5-26: EBSD phase maps from the core cross-section (500-750 microns from the surface) of G3 alloy as a function of true strain.

5.2.5 Fractography

The fracture surfaces of the carbon graded steels were examined after uniaxial tensile testing to observe features present in the damage zone and determine the main fracture mode. The fractography results for the G1 alloy were similar to those of the G2 alloy and are presented in Appendix C. Figure 5-27 illustrates the SEM micrographs from the fracture surface of the G2 alloy. Micro-cracks, micro-voids, cleavage facets and

ductile tearing features can be observed. However, the near-surface layer (Figure 5-27(a)) contained a higher area fraction of cleavage facets and less ductile tearing features than the mid-thickness and core layers (Figure 5-27(b) and (c)). Moreover, the size and number of both micro-cracks and micro-voids decreased from the surface towards the core. Thus, the dominant fracture mode was concluded to become more ductile from the near-surface layer towards the core layer. However, the cleavage characteristics of fracture surface for the near-surface cross-section were found to control the fracture of the G2 alloy as these features usually accompany low uniform elongations (Koyama et al., 2011, Lee et al., 2014, Liang, 2008, Yang, 2010). This can explain the relatively low uniform elongation observed for this alloy versus homogenous 0.6C.



(a) near surface (0-250 μ m from the surface)

(b) mid-thickness (250-500 µm from the





(c) core (500-750 μ m from the surface)

Figure 5-27: SEM micrographs from fracture surface of G2 alloy.

The sample cross-sections near the fracture surface, sectioned along the tensile direction, of the carbon graded steels were also examined after uniaxial tensile testing to identify the main damage mechanism. Figure 5-28 displays typical SEM micrographs from the cross-section near the fracture surface of the G2 alloy. In the near surface cross-section layer of the fracture sample, micro-voids and cracks were frequently observed along austenite/ɛ-martensite interfaces, as illustrated in Figure 5-28(a). Void nucleation at austenite/ɛ-martensite interfaces was found to be the dominant damage mechanism for the

near-surface layer of the G2 alloy. This was consistent with other studies in the literature concerning *ɛ*-martensite formation dominated alloys tensile tested to failure (Koyama et al., 2011, Lee et al., 2014, Liang, 2008, Yang, 2010). Since this damage mechanism results in a relatively low uniform elongation (Koyama et al., 2011, Lee et al., 2014, Liang, 2008, Yang, 2010), it was determined as the root cause for the global fracture of the G2 alloy. This is consistent with the relatively high volume fraction of ε -martensite observed within the near surface cross-section layer of G2 alloy at fracture (Figure 5-19). In the mid-thickness cross-section layer of the fracture sample (Figure 5-28(b)), significant occurrences of void nucleation at grain boundaries were observed, indicating that this was the dominant damage mode for this cross-section layer of the G2 alloy. In the core cross-section of the alloy (Figure 5-28(c)), decohesion of a series of inclusions was observed in addition to void nucleation at grain boundaries. Thus, the damage mode for this layer of the G2 alloy was concluded to be void nucleation at grain boundaries and inclusion/matrix interfaces. However, this damage mode was determined not to be the critical damage mode for the G2 alloy because it cannot explain the lower uniform elongation of this alloy versus the homogenous 0.6C alloy.



(a) near surface cross-section (0-250 μ m from

the surface)

(b) mid-thickness cross-section (250-500 µm



(c) core cross-section (500-750 μ m from the surface)

Figure 5-28: SEM micrographs from cross-section near fracture surface, sectioned along tensile direction, of G2 alloy.

The SEM micrographs from the fracture surface of the G3 alloy are illustrated in Figure 5-29. Fracture features comprised micro-voids, micro-cracks, cleavage facets and ductile tearing features. The near surface layer (Figure 5-29(a)) contained more cleavage facets and less ductile tearing features than the mid-section layer (Figure 5-29(b)) and core layer (Figure 5-29(c)). Moreover, compared to the G2 alloy (Figure 5-27), here the mid-section and core layers contained more cleavage facets and less ductile tearing

from the surface)

McMaster – Materials Science & Engineering.

features (Figure 5-29(b) and (c)). This can be attributed to deformation products within the cross-section of this alloy at high strains detected by the EBSD technique (Figure 5-24 to Figure 5-26), where the depth into the sample for which mechanical ε -martensite was the dominant deformation product was greater for the G3 steel versus the G1 and G2 alloys. From the fractographs presented in Figure 5-29, it can be concluded that the fracture surface of the G3 alloy had predominantly cleavage characteristic with some ductile fracture characteristic at the core layer. This led to the relatively low uniform elongation for this alloy compared to the homogenous 0.6C alloy. The correlation between the cleavage characteristic of the fracture surface and the relatively low uniform elongation is well established in the literature (Koyama et al., 2011, Lee et al., 2014, Liang, 2008, Yang, 2010).



(a) near surface (0-250 μ m from the surface)

(b) mid-section (250-500 μ m from the surface)



(c) core (500-750 μ m from the surface)

Figure 5-29: SEM micrographs from fracture surface of G3 alloy.

The typical SEM micrographs from cross-sections near the fracture surface, sectioned along the tensile direction, of the G3 alloy are presented in Figure 5-30. In the near surface and mid-section cross-section layers (Figure 5-30(a) and (b)), micro-voids and cracks were frequently observed along austenite/ɛ-martensite interfaces. Void nucleation at austenite/ɛ-martensite interface was found to be the dominant damage mechanism for the near surface and mid-section cross-section layers of the G3 alloy. This damage mechanism was also concluded to be the main cause for the global fracture behaviour of the G3 alloy. In this way, the relatively low uniform elongation of the G3

alloy versus the homogenous 0.6C alloy can be well explained. The same damage mechanism was already reported for several ε-martensite formation dominated alloys tensile tested to fracture (Koyama et al., 2011, Lee et al., 2014, Liang, 2008, Yang, 2010). In the core cross-section of the G3 alloy (Figure 5-30(c)), decohesion of a series of inclusions of different sizes was observed as well as void nucleation at grain boundaries. Thus, the damage mode for this cross-section layer of G3 alloy was determined to be void nucleation at grain boundaries and inclusion/matrix interfaces. However, this fracture mechanism did not dominate the global fracture of the G3 alloy.


(a) near surface cross-section (0-250 μ m from



(b) mid-thickness cross-section (250-500 µm

from the surface)



(c) core cross-section (500-750 μ m from the surface)

Figure 5-30: SEM micrographs from cross-section near fracture surface, sectioned along tensile direction, of G3 alloy.

5.2.6 Summary of Carbon Graded Fe-22Mn-C Alloys

For all the carbon graded alloys, the as heat-treated microstructure near the surface cross-section layer was a mixture of austenite and thermal ε -martensite, where the ε -martensite volume fraction decreased towards the core. The as heat-treated microstructure of the core cross-section layer for all steels comprised an austenite matrix with some annealing twins. For all alloys, the dominant deformation products altered

the surface)

from mechanical ε -martensite at the near-surface cross-section layer to a mixture of mechanical twins and ε -martensite or only mechanical twins at the inner cross-section layers.

The G3 alloy exhibited a higher initial work hardening rate but a lower uniform elongation than the other two steels because the depth into the sample for which ε-martensite was the dominant deformation product was greater for this alloy. The flow curves of the G1 and G2 alloys displayed serrations, which was attributed to the Portevin-LeChatlier (PLC) or dynamic strain aging (DSA) effect.

The formation of both the mechanical twins and mechanical ε -martensite started at a specific true strain and saturated at high strains, resulting in the sigmoidal-shape for the volume fraction plots as a function of true strain.

6 EXPERIMENTAL RESAULTS FOR FE-30MN-C ALLOYS

In this chapter, the experimental results for both the homogenous and carbon graded Fe-30Mn-C alloys will be presented. The homogenous alloy results will be presented in §6.1 followed by the carbon graded alloy results in §6.2. A detailed discussion of the experimental results can be found in Chapter 7.

6.1 Homogenous Fe-30Mn-C Alloys

In this section, the carbon content and microstructure of the homogenous as heattreated alloys will be presented, followed by results concerning the mechanical properties and microstructural evolution during tensile deformation. Finally, a fracture analysis of all homogenous Fe-30Mn-C alloys will be presented.

6.1.1 As Heat-Treated Alloy Carbon Content and Microstructure

The as-received Fe-30Mn-0.6C steel was heat treated following the methods mentioned in Chapter 4 to obtain three homogenous steels with lower carbon contents. The carbon contents of the as heat-treated homogenous Fe-30Mn-C alloys as well as the stacking fault energy (SFE) values determined using available models can be found in Table 6-1. For all the alloys except the 0C alloy, the measured carbon contents were very close to the target levels. According to the SFE model of Dumay et al. (Dumay et al., 2008), the deformation products were expected to be mechanical twins for the 0.2C, 0.4C and 0.6C alloys and a combination of mechanical twins and mechanical ε -martensite for the 0C alloy. This model also predicted a fully austenitic microstructure (without any thermal ε -martensite) for all the as heat-treated alloys. The SFE model developed by

Nakano and Jacques (Nakano and Jacques, 2010) predicted mechanical ε-martensite formation for the 0C alloy without providing any comments on mechanical twinning. This model predicted a fully austenitic microstructure (with no thermal ε-martensite) for all the as heat-treated alloys too. The SFE model proposed by Saeed-Akbari et al. (Saeed-Akbari et al., 2009) did not predict mechanical ε-martensite formation for any of the experimental alloys. As was seen in the Nakano and Jacques model (Nakano and Jacques, 2010), this model does not contain a SFE range for mechanical twinning. Moreover, it should be noted that the results listed in Table 6-1 indicate that the Dumay et al. model (Dumay et al., 2008) contains a stronger effect of the alloy carbon content on the SFE value compared to the other models.

Table 6-1. Carbon content and computed SFE values for all homogenous Fe-30Mn-C alloys.

Alloy	Carbon content	SFE (mJ/m^2) by	SFE (mJ/m ²) by	SFE (mJ/m^2) by
Name	(wt. %)	(Dumay et al., 2008)	(Nakano and	(Saeed-Akbari et al.,
			Jacques, 2010)	2009)
0C	0.06	17.9	35.3	28.4
0.2C	0.20	23.5	37.8	31.1
0.4C	0.41	29.6	40.6	35.1
0.6C	0.59	33.2	43.4	38.5

The average grain size of the as heat-treated samples was $104 \pm 4 \mu m$, $101 \pm 2 \mu m$, $110 \pm 3 \mu m$ and $108 \pm 4 \mu m$ for the 0C, 0.2C, 0.4C and 0.6C alloys, respectively. From this data, it can be concluded that there were only minor differences in the grain sizes of the alloys. As was the case for the homogenous 22Mn alloys, EBSD was used for the microstructural analysis of all alloys. For all EBSD phase maps presented, it should be

noted that FCC austenite is red, HCP ε-martensite blue, twin boundaries in yellow and un-indexed areas in black. Moreover, grain boundaries are highlighted as black lines.

Figure 6-1 illustrates typical EBSD phase maps from the cross-sections of the as heat-treated homogenous alloys. For all the alloys, the microstructures comprised an equiaxed austenitic matrix with some annealing twins and no detectable ε -martensite. This arose from the high manganese content of the experimental alloys increasing the SFE. For all homogenous alloys, the as heat-treated microstructures generally confirmed the predictions of the Dumay et al. (Dumay et al., 2008) and Nakano and Jacques (Nakano and Jacques, 2010) SFE models.



(c) 0.4C

(d) 0.6C

Figure 6-1: Typical EBSD phase maps from the cross-section of as heat-treated homogenous Fe-30Mn-C alloys.

6.1.2 Tensile Behaviour

In the present sub-section, the tensile behaviour of homogenous Fe-30Mn-C alloys at room temperature will be presented. For each experimental alloy, a minimum of three uniaxial tensile tests were performed to assess the repeatability of the results. True stress-true strain curves of the as heat-treated 0C alloy at room temperature are presented in Figure 6-2. From this, it can be seen that the results of the three uniaxial tensile tests

were consistent. For later comparison and discussion, the results of test 1 were selected as being representative of the tensile behaviour for the 0C alloy. The tensile results for the other alloys were similarly repeatable; a complete compilation of which can be found in Appendix A. For all other alloys, a representative tensile curve was selected for further discussion.



Figure 6-2: Room temperature true stress-true strain curves for the homogenous Fe-30Mn-0C alloy.

Representative room temperature true stress-true strain curves for all homogenous Fe-30Mn-C alloys as a function of alloy C content are shown in Figure 6-3(a) and the derived tensile properties for all alloys listed in Table 6-2. All tensile curves exhibited continuous yielding. For all the homogenous alloys, it can be seen that the yield stress increased with increasing carbon content (Figure 6-3(a), Table 6-2). This can be explained by the solid solution hardening effect of carbon in austenite (Bouaziz et al., 2011b, Ohkubo et al., 1994).



(a) Representative true stress-true strain curves as a function of alloy C content.



(b) Corresponding work hardening rate versus true stress as a function of alloy C content.

Figure 6-3: Room temperature tensile response of homogenous Fe-30Mn-C alloys.

Alloy Name	Yield Stress (MPa)	UTS (MPa)	Uniform Elongation
0C	153 ± 3	775 ± 7	0.48 ± 0.03
0.2C	174 ± 4	892 ± 8	0.47 ± 0.02
0.4C	251 ± 6	1165 ± 10	0.50 ± 0.03
0.6C	286 ± 5	1457 ± 9	0.61 ± 0.04

Table 6-2. Room temperature tensile characteristics of homogenous Fe-30Mn-C alloys.

It can also be seen from Table 6-2 that the ultimate tensile strength (UTS) increased with increasing alloy carbon content, which can be attributed to the sustained high work hardening rates exhibited by these alloys as well as the solid solution hardening effect of carbon (Figure 6-3(b)). The uniform elongation of the 0.6C alloy was significantly higher compared the other alloys, which was attributed to the highest sustained work hardening rate observed for this alloy. The UTS and uniform elongation values of the 0C alloy were higher than previously reported for this alloy (Liang et al., 2009). The discrepancy in the uniform elongation can be explained by the larger grain size (104 μ m) of this alloy prior to tensile testing versus 20-50 μ m (Liang et al., 2009). The difference in the UTS value, however, arose from the actual carbon content of this alloy (0.06 wt. %, see Table 6-2) versus 0.02 wt. % (Liang et al., 2009), which can increase the UTS through increasing the work hardening rate in addition to the solid solution hardening. The flow curves of the 0.4C and 0.6C alloys showed significant serrations, attributed to the Portevin-LeChatelier (PLC) or dynamic strain aging (DSA) effect (Figure 6-3(a)) (Chen et al., 2007, Renard et al., 2010). Serrations were not observed in the flow curves of the 0C and 0.2C alloys. However, these alloys had a lower work hardening rate than the 0.4C and 0.6C alloys (Figure 6-3(b)), which was continuously declining with increasing stress and strain. As shown by Liang et al. (Liang deformation mode was dislocation glide.

Further inspection of Figure 6-3(b) shows that the experimental alloys exhibited distinctly different work hardening histories, where the 0C and 0.2C alloys showed relatively low work hardening (i.e. $d\sigma/d\epsilon$) values during plastic deformation which continuously declined with increasing strain. These work hardening histories are consistent with those observed by Liang et al. (Liang et al., 2009) for a Fe-30Mn-0C alloy, in which dislocation glide was the dominant deformation mode. However, the 0.4C and 0.6C alloys showed several distinct stages to their $d\sigma/d\epsilon$ values, where the $d\sigma/d\epsilon$ values for the 0.4C alloy initially declined with increasing strain until $\sigma \approx 570$ MPa ($\varepsilon \approx$ 0.15) before attaining a maximum at $\sigma \approx 890$ MPa ($\varepsilon \approx 0.33$), after which the work hardening rates declined to fracture (Figure 6-3(b)). Similarly, the $d\sigma/d\epsilon$ values for the 0.6C alloy initially declined with increasing strain until $\sigma \approx 660$ MPa ($\epsilon \approx 0.18$), after which they increased until $\sigma \approx 1050$ MPa ($\epsilon \approx 0.38$), followed by declining work hardening rates to fracture. This behaviour is consistent with stages A through C of the work hardening curves of a Fe-20Mn-1.2C TWIP steel as described by Renard and Jacques (Renard and Jacques, 2012) and is consistent with the work hardening behaviour generally observed for high-Mn TWIP steels (Chen et al., 2007, Barbier et al., 2009, Renard et al., 2010, Yang et al., 2010), including the 22Mn TWIP steels of the present study.

6.1.3 Microstructural Evolution with Tensile Deformation

To determine the products in the deformed microstructures, EBSD and XRD analyses were performed on homogenous alloys as a function of plastic strain. A plot of ε -martensite volume fraction as a function of true strain for the homogenous Fe-30Mn-OC alloy is depicted in Figure 6-4. For the un-deformed microstructure (at $\varepsilon = 0$), the ε -martensite volume fraction was approximately zero. This was consistent with the microstructural observation of the as heat-treated OC alloy illustrated in Figure 6-1(a). It can be seen that the ε -martensite volume fraction increased in a roughly sigmoidal fashion with increasing true strain, indicating that austenite transformed to mechanical ε -martensite during deformation. It can also be seen that the rate- ω f martensitic transformation declined for $\varepsilon \ge 0.3$. These observations are reasonably consistent with the 22Mn ε -martensite forming alloys in the present study.



Figure 6-4: Evolution of ε -martensite volume fraction with tensile strain for the homogenous Fe-30Mn-0C alloy, measured via XRD.

Figure 6-5 illustrates EBSD phase maps from the cross-sections of the deformed OC alloy. At low strain levels (Figure 6-5(a), (b) and (c)), similar to the as heat-treated steel (Figure 6-1(a)), the microstructure comprised an equiaxed austenite matrix and some annealing twins. At higher strain levels (Figure 6-5(d), (e) and (f)), the deformation products were small volume fractions of both mechanical twins and ε -martensite. The latter observation was consistent with the XRD data in Figure 6-4, which showed that there was a small but significant volume fraction of mechanical ε -martensite within the microstructure for $\varepsilon \ge 0.05$. However, the detected ε -martensite volume fraction (≈ 0.12) was not large enough to determine that ε -martensite was the dominant deformation product for this alloy.



(a) ε=0.02



(b) ε=0.05





(d) ε=0.2



Figure 6-5: EBSD phase maps of cross-sections of the 0C alloy as a function of true strain.

The point counting method of Renard and Jacques (Renard and Jacques, 2012) was used to estimate the volume fraction of twins based on the EBSD phase maps. Figure 6-6 depicts the evolution of twin volume fraction with true strain for all the homogenous Fe-30Mn-C alloys. It can be seen that for all experimental alloys, the twin volume fraction was approximately zero when true strain was lower than 0.1. This implied that no mechanical twins formed within the microstructures in low applied strains. In the case of 0C alloy, the volume fraction of twins increase from $\varepsilon = 0.1$ to necking, though the curve saturated at high strains. However, the saturated twin volume fraction for this alloy was not large enough to state that mechanical twins were determined to be the dominant deformation products for this alloy.

From both the EBSD phase maps (Figure 6-5) and XRD data (Figure 6-4), the dominant deformation mode for the 0C alloy was concluded to be dislocation glide, though small volume fractions of both the mechanical twins and mechanical ε -martensite were detected as deformation products. This is similar to the deformation products reported by Liang et al. for a Fe-30Mn-0C alloy when deformation was made through uniaxial tension at 293 K (Liang et al., 2009). The mixed deformation products were also observed for the Fe-22Mn-0.4C alloy in the present study, where the SFE for that alloy (15.0 mJ/m²) was not significantly different from that of the Fe-30Mn-0C alloy (17.9 mJ/m²). However, a larger volume fraction of mechanical twins was observed upon deformation of the 22Mn-0.4C alloy versus the 30Mn-0C alloy.



Figure 6-6: Twin volume fraction as a function of strain for all the homogenous Fe-30Mn-C alloys.

Figure 6-7 illustrates EBSD phase maps from the cross-sections of the deformed 0.2C alloy. For low strain levels (Figure 6-7(a), (b) and (c)), the microstructure consisted of a fully austenitic matrix with some annealing twins, which was similar to the as heat-treated microstructure (Figure 6-1(b)). As the true strain approached $\varepsilon = 0.2$ (Figure 6-7(d)), mechanical twins were first observed. Then the twin volume fraction increased sigmoidally with increasing deformation until necking with a trend similar to that observed for the 0C alloy (Figure 6-6). It should be noted, however, that the twin volume fraction at higher strains was higher versus the 0C alloy. This explained the higher work

hardening rate observed for the 0.2C alloy compared to the 0C alloy, which led to higher UTS (Figure 6-3). However, the increasing twin volume fraction of the 0.2C alloy was not large enough to prevent a continuous decease in the work hardening rate during the deformation of this alloy, as observed in Figure 6-3(b). No ε -martensite was observed in the microstructure of this alloy for all applied strains, as shown in Figure 6-7. Thus, it can be concluded that dislocation glide was the dominant deformation mode for this alloy. This caused tensile behaviour of this alloy to resemble that of the 0C alloy (see Figure 6-3), which was previously determined to deform primarily through dislocation glide (Liang et al., 2009).



(a) ε=0.02



(b) ε=0.05



(c) ε=0.1



(d) ε=0.2



Figure 6-7: EBSD phase maps of cross-sections for 0.2C alloy as a function of true strain.

EBSD phase maps from the cross-sections of the deformed 0.4C alloy are presented in Figure 6-8. For true strains (ε) up to 0.1 (Figure 6-8(a)-(c)), similar to the as heat-treated steel (Figure 6-1(c)), the microstructure was composed of an austenite matrix and some annealing twins. As the true strain approached 0.2 (Figure 6-8(d)), mechanical twins started to develop within the microstructure. Then twin population increased with increasing deformation until necking (Figure 6-8(e) and (f)). From Figure 6-6, it can be seen that the volume fraction of twins increased sharply in the true strain range of 0.1-0.3. Further deformation led to a slight increase of twin volume fraction until necking. It should also be noted that the twin volume fraction at higher strains was significantly higher versus that of the 0C and 0.2C alloys. This caused the work hardening behaviour of the 0.4C alloy to resemble that of a typical TWIP steel.

Further examination of Figure 6-8 reveals that the twin population for different grains was not the same, which is consistent with general findings in the literature concerning the effect of the local Schmid factor on twin activation (Allain et al., 2002, Barbier et al., 2009, Gutierrez-Urrutia et al., 2010, Yang et al., 2006). Based on the EBSD phase maps observed in Figure 6-8, the dominant deformation product for the 0.4C alloy was determined to be mechanical twins. This is similar to the deformation products reported by Wang et al. for a Fe-30Mn-0.5C alloy tensile tested at room temperature (Wang et al., 2010) and for the 22Mn-0.6C alloy in the present study.



(a) ε=0.02



50 μm

(c) ε=0.1

(b) ε=0.05



(d) ε=0.2



Figure 6-8: EBSD phase maps of cross-sections for 0.4C alloy as a function of true strain.

50 µm



Figure 6-9 illustrates EBSD phase maps from the cross-sections of the deformed 0.6C alloy. For low strain levels (Figure 6-9(a)-(c)), the microstructure comprised an austenitic matrix with some annealing twins. Mechanical twins were first observed when the true strain reached 0.2 (Figure 6-9(d)). For higher strain levels (Figure 6-9(e) and (f)), the mechanical twin population increased with increasing deformation. As it can be seen from Figure 6-6, the twin volume fraction increased with increasing deformation until necking with a trend similar to that observed for the other homogenous Fe-30Mn-C alloys. However, it should be noted that the twin volume fraction at higher strain for this alloy was the highest among the homogenous Fe-30Mn-C alloys. This resulted in the alloy exhibiting the highest work hardening rate among all the homogenous Fe-30Mn-C alloys (Figure 6-3(b)), considering the so called "dynamic Hall-Petch effect" role of the twins in refining the microstructure and, thereby, reducing the mean free path for dislocation motion (Allain et al., 2004a, Bouaziz and Guelton, 2001, Shiekhelsouk et al., 2009). According to the EBSD phase maps observed in Figure 6-9, the dominant deformation product for the 0.6C alloy was found to be the mechanical twins. This confirmed the predictions of the Dumay et al. SFE model (Dumay et al., 2008).





Figure 6-9: EBSD phase maps of cross-sections for 0.6C alloy as a function of true strain.

McMaster – Materials Science & Engineering.

6.1.4 Fractography

In order to observe features presented in the damage zone and identify the main fracture mode for all homogenous alloys, the fracture surfaces and sample cross-sections near the fracture surfaces were examined after uniaxial tensile testing to failure. Figure 6-10 illustrates the SEM micrographs from the fracture surface ((a) and (b)) and the crosssection near the fracture surface (c), sectioned along the tensile direction, for the Fe-30Mn-0C alloy. The fracture surface displayed micro-cracks, micro-voids, a large area fraction of ductile tearing features and some cleavage facets. The relative population of these features is in agreement with the small volume fractions of ε -martensite detected by XRD and EBSD analyses in the fractured sample microstructures, as shown in Figure 6-4 and Figure 6-5, respectively. Ductile fracture was then determined to be the dominant fracture mode for this alloy. In the cross-section of the fracture sample, decohesion of inclusions at the austenite/ ϵ -martensite interfaces and grain boundaries were frequently observed, as illustrated in Figure 6-10(c), indicating that these were the dominant damage mechanism for this alloy. Similar fractography results were reported by Liang (Liang, 2008) for an Fe-30Mn-0C alloy tensile tested to failure at 293 K.



(a) low magnification view

(b) higher magnification view



(c) decohesion of inclusion at the austenite/ɛ-martensite interface

Figure 6-10: SEM fractographs of homogenous Fe-30Mn-0C alloy.

Figure 6-11 displays the SEM micrographs from the fracture surface ((a) and (b)) and the cross-section near the fracture surface (c), sectioned along the tensile direction, for the Fe-30Mn-0.2C steel. Various fracture features observed include micro-cracks, micro-voids, a large area fraction of ductile tearing features and a small area fraction of cleavage facets (Figure 6-11(a) and (b)). Thus, the dominant fracture mode for this alloy was determined to be ductile fracture. In the cross-section of the fracture sample, micro-voids and cracks were observed at the grain-boundaries and also inside the grains at twin-

austenite boundaries, as displayed in Figure 6-11(c), indicating that these were significant sites for damage nucleation and void growth.



(a) low magnification view

(b) higher magnification view



(c) void nucleation at a grain boundary

Figure 6-11: SEM fractographs of homogenous Fe-30Mn-0.2C alloy.

Figure 6-12 illustrates the SEM micrographs from the fracture surface ((a) and (b)) and the cross-section near the fracture surface (c), sectioned along the tensile direction, for the homogenous Fe-30Mn-0.4C steel. The fracture features mainly comprised micro-voids, micro-cracks and ductile tearing features with no significant cleavage facets being observed (Figure 6-12(a) and (b)). Thus, for the Fe-30Mn-0.4C

alloy, it can be concluded that the fracture mode was predominately ductile. In the crosssection of the alloy near the fracture surface (Figure 6-12(c)), significant occurrences of twin boundary/matrix separation and void nucleation at inclusions were observed, indicating that these were the dominant damage modes for this alloy.



(a) low magnification view

(b) higher magnification view



(c) crack, void and inclusion at a grain boundary

Figure 6-12: SEM fractographs of homogenous Fe-30Mn-0.4C alloy.

Figure 6-13 displays the SEM micrographs from the fracture surface ((a) and (b)) and the cross-section near fracture surface (c), sectioned along the tensile direction, for the Fe-30Mn-0.6C steel. In the fracture surface (Figure 6-13(a) and (b)), micro-voids,

micro-cracks and ductile tearing features were observed with no significant cleavage facets being observed. Thus, ductile fracture was determined to be the dominant fracture mode for this steel. In the fracture cross-sections of the alloy (Figure 6-13(c)), decohesion of a series of inclusions along a grain boundary was observed in addition to void nucleation at grain boundaries. Thus, the damage mode for this alloy was concluded to be void nucleation at grain boundaries and inclusion/matrix interfaces.



(a) low magnification view

(b) higher magnification view



(c) decohesion of inclusions

Figure 6-13: SEM fractographs of homogenous Fe-30Mn-0.6C alloy.

6.1.5 Summary of Homogenous Fe-30Mn-C Alloys

For all the homogenous Fe-30Mn-C alloys, the as heat-treated microstructure was composed of an equiaxed austenite matrix with some annealing twins. Upon deformation, the microstructure of the 0C alloy consisted of austenite and small volume fractions of both mechanical ε-martensite and twins as deformation products, resulting in the alloy displaying a low work hardening rate typical of softer FCC metals. The deformed microstructure of the 0.2C alloy revealed a small volume fraction of mechanical twins as the deformation products, with dislocation glide being the dominant deformation mode for this alloy. The deformation products for both the 0.4C and 0.6C alloys were found to be significant volume fractions of mechanical twins, leading to the alloys exhibiting high sustained work hardening rates.

The yield stress and tensile strength of homogenous Fe-30Mn-C alloys increased with increasing alloy C level whereas the uniform elongation did not change significantly, except for the 0.6C alloy.

6.2 Carbon Graded Fe-30Mn-C Alloy

In this section, the experimental results for the carbon graded Fe-30Mn-C alloy will be presented. The carbon concentration profile and the microstructure of the as decarburized alloy will be presented first, followed by results concerning the tensile behaviour and microstructural evolution during tensile deformation. Finally, a fracture analysis of the carbon graded Fe-30Mn-C alloy will be presented.

6.2.1 As Decarburized Alloy Carbon Concentration Profile and SFE

As-received Fe-30Mn-0.6C steel was decarburized following the method mentioned in Chapter 4 to obtain the desired carbon gradient. A carbon concentration profile from the surface to the core for the graded alloy is depicted in Figure 6-14. The carbon concentration profile was assumed to be symmetric across the centre plane of the sample. This assumption was verified by microstructural observation, where the observed edge was not necessarily the same as the one already used for carbon concentration increased from 0.12 wt. % at the surface to 0.55 wt. % at the core. The relatively low carbon concentration at the surface can be attributed to the utilization of wet hydrogen as the decarburization agent.



Figure 6-14: Carbon concentration profile for the carbon graded Fe-30Mn-C steel. Note that the total sheet thickness was 1.6 mm or 1600 microns and that symmetry was assumed.

A manganese concentration profile from the surface to the core for the carbon graded Fe-30Mn-C alloy is depicted in Figure 6-15. The manganese concentration at the surface layer (0-100 microns from the surface) was lower versus the inner cross-section layers because manganese oxidized during decarburization heat-treatment. For the inner cross-section layers (100-800 microns from the surface), the manganese concentration did not change significantly with sample depth, being approximately 30 wt. %. This value was very close to the bulk manganese content (30.28 ± 0.68 wt. %) of the as-received Fe-30Mn-0.6C steel (see Table 4-1).



Figure 6-15: Manganese concentration profile for the carbon graded Fe-30Mn-C alloy.

Using the carbon concentration profile presented in Figure 6-14, the alloy SFE values as a function of sample depth were calculated based on the Dumay et al. (Dumay et al., 2008) SFE model for the carbon graded Fe-30Mn-C steel. The resultant plot is depicted in Figure 6-16. For the carbon graded steel and the resultant SFE gradient, the as-decarburized microstructure was expected to be fully austenitic (with no thermal ε -martensite) even near the outer surface (Dumay et al., 2008). For this alloy, it was further expected that the deformed microstructure would exhibit mixed ε -martensite/twin deformation products at the outer surface and would mechanically twin at the core (Dumay et al., 2008).



Figure 6-16: SFE versus sample depth for carbon graded Fe-30Mn-C steel based on the Dumay et al. (Dumay et al., 2008) SFE model.

6.2.2 As Heat-Treated Microstructure

EBSD was used for the microstructural analysis of the graded Fe-30Mn-C alloy. For all EBSD phase maps presented, FCC austenite is denoted in red, HCP ε -martensite blue, FCC manganese oxide (MnO) green, BCT α '-martensite white and un-indexed areas black. Grain boundaries are highlighted as black lines and twin boundaries in yellow.

Figure 6-17 illustrates the EBSD phase maps from the cross-section of the as decarburized carbon graded Fe-30Mn-C steel. A continuous external MnO layer was present (Figure 6-17(a)), arising from the selective oxidation of manganese during the decarburization process. This layer was partially removed from the steel surface during

Ph.D. Thesis – M. Ghasri Khouzani McMaste

McMaster – Materials Science & Engineering.

metallographic preparation. There was also a thermal α '-martensite layer observed immediately below the MnO external film, in which some MnO particles were observed. The manganese content of the α '-martensite layer was measured by EDS spot analysis to be approximately 8 wt. %. This indicates that the α '-martensite formed due to manganese depletion of the surface layer. Meguerian (Meguerian, 2007) has previously reported the formation of thermal α '-martensite for similar Mn and C levels. A relatively thin layer of thermal ε -martensite was also observed just below the α '-martensite layer (Figure 6-17(a)), where the formation of this layer can be attributed to manganese depletion.

Further examination of Figure 6-17 shows that for the as heat-treated carbon graded Fe-30Mn-C steel, the microstructure comprised austenite with some annealing twins. This was consistent with predictions of the Dumay et al. (Dumay et al., 2008) SFE model.





(c) 500-750 μ m from surface

Figure 6-17: EBSD phase maps from cross-section of as decarburized carbon graded Fe-30Mn-C steel.

6.2.3 Tensile Behaviour

In the present sub-section, the tensile behaviour of the carbon graded Fe-30Mn-C steel at room temperature will be presented. For comparison purposes, the tensile behaviour of the monolithic 0.6C steel (see Chapter 4), will also be presented. For each experimental alloy, a minimum of three uniaxial tensile tests were performed to assess the

Ph.D. Thesis – M. Ghasri KhouzaniMcMaster – Materials Science & Engineering.consistency of the results. The tensile results for both the alloys were repeatable, acomplete compilation of which can be found in Appendix A.

Typical room temperature true stress-true strain (σ - ϵ) plots for both the carbon graded Fe-30Mn-C and monolithic Fe-30Mn-0.6C steels are presented in Figure 6-18(a) and their tensile properties summarized in Table 6-3. Both tensile curves exhibited continuous yielding. The carbon graded alloy had a lower yield stress relative to the monolithic steel. This can be explained by the solid solution hardening effect of carbon in austenite (Bouaziz et al., 2011b, Ohkubo et al., 1994) when considering the lower overall carbon content of the as decarburized graded alloy (Figure 6-14) compared to the asannealed monolithic alloy (0.59 wt. % C).

The flow curves of both the carbon graded and monolithic alloys showed significant serrations, attributed to the Portevin-LeChatelier (PLC) or dynamic strain aging (DSA) effect (Figure 6-18(a)) (Chen et al., 2007, Renard et al., 2010). This indicates that these alloys had sufficient overall carbon content to promote the PLC effect.



(b) Corresponding work hardening rate as a function of true stress

Figure 6-18: Tensile response of carbon graded and monolithic alloys at RT.
Table 6-3. Room temperature tensile characteristics of the carbon graded Fe-30Mn-C and monolithic Fe-30Mn-0.6C alloys.

Alloy Name	Yield Stress (MPa)	UTS (MPa)	Uniform Elongation
Graded	209 ± 3	1111 ± 8	0.53 ± 0.03
Monolithic	278 ± 5	1450 ± 10	0.62 ± 0.04

Further inspection of Figure 6-18(b) shows that the monolithic alloy exhibited a higher sustained work hardening rate versus the carbon graded alloy during plastic deformation. This resulted in the monolithic alloy displaying higher UTS and larger uniform elongation (Figure 6-18(a) and Table 6-3). Moreover, both the alloys showed several distinct stages to their $d\sigma/d\epsilon$ values, where the $d\sigma/d\epsilon$ values for the graded alloy initially declined with increasing strain until $\sigma \approx 540$ MPa ($\varepsilon \approx 0.15$) before attaining a maximum at $\sigma \approx 880$ MPa ($\epsilon \approx 0.36$), after which the work hardening rates declined to fracture (Figure 6-18(b)). Similarly, the $d\sigma/d\epsilon$ values for the monolithic alloy initially declined with increasing strain until $\sigma \approx 660$ MPa ($\varepsilon \approx 0.18$), after which they increased until $\sigma \approx 1050$ MPa ($\epsilon \approx 0.38$), followed by declining work hardening rates to fracture. This behaviour is consistent with the work hardening curves of the 0.4C and 0.6C alloys, as shown in Figure 6-3(b) and is consistent with the work hardening behaviour generally observed for high-Mn TWIP steels (Chen et al., 2007, Barbier et al., 2009, Renard and Jacques, 2012, Renard et al., 2010, Yang et al., 2010) including the homogenous 22Mn-0.4C and 22Mn-0.6C alloys in this study.

6.2.4 Microstructural Evolution with Tensile Deformation

To determine the deformation products in the deformed microstructures, EBSD analyses were performed on the carbon graded steel as a function of plastic strain. XRD analyses were also attempted in order to evaluate the microstructural evolution with tensile strain; however, the results were not useful due to apparatus limitations. Figure 6-19 illustrates the EBSD phase maps from the near surface cross-section (0-250 µm from the surface) of the carbon graded Fe-30Mn-C alloy as a function of strain. At $\varepsilon = 0.15$ (Figure 6-19(a)), the near-surface microstructure of the carbon graded steel was composed of an austenite matrix with some annealing twins, which was similar to the as decarburized microstructure (Figure 6-17(a)). At higher strain levels (Figure 6-19(b)-(d)), the deformation products were mechanical twins and ε -martensite, where the volume fraction of these products increased with deformation. This confirmed the predictions of Dumay et al. (Dumay et al., 2008) SFE model, where the SFE for this surface layer changed from 20.4 mJ/m² to 29.2 mJ/m² (see Figure 6-16). Similar deformation products were observed for the present homogenous 30Mn-0C alloy (Figure 6-5).



(c) ε=0.3

(d) ε=0.45

Figure 6-19: EBSD phase maps from the near surface cross-section (0-250 microns from the surface) of carbon graded alloy as a function of true strain.

50 µm

Figure 6-20 shows the evolution of the twin volume fraction with true strain based on the point counting method. For the near-surface layer of the carbon graded alloy, the volume fraction of twins increased slightly up to ≈ 0.15 . Fur ther deformation led to a higher increase of twin volume fraction until necking with a trend similar to that observed for the homogenous alloys (Figure 6-17). Moreover, the saturated volume fraction of the mechanical twins for the surface layer was similar to the one observed for the homogenous 0.2C alloy.



Figure 6-20: Twin volume fraction as a function of strain for the carbon graded alloy.

The EBSD phase maps from the mid-thickness cross-section (250-500 microns from the surface) of the carbon graded Fe-30Mn-C alloy as a function of applied strain are displayed in Figure 6-21. At a true strain (ϵ) of 0.15 (Figure 6-21(a)), similar to the as decarburized steel (Figure 6-17(b)), the microstructure of the mid-thickness layer comprised an equiaxed austenitic matrix with some annealing twins. As the true strain approached 0.2 (Figure 6-21(b)), mechanical twins were first observed, where the twin volume fraction sigmoidally increased with increasing deformation, as shown in Figure

Ph.D. Thesis – M. Ghasri Khouzani

McMaster – Materials Science & Engineering.

6-20. It should be noted, however, that the twin volume fraction at higher strains was significantly higher versus the near-surface cross-section layer. This can be explained by considering the SFE profile of this steel (Figure 6-16), where the SFE increased to 29.2- 31.9 mJ/m^2 for the mid-thickness layer. Although the average SFE value for the mid-thickness layer was very similar to that of the homogenous 0.4C alloy – i.e. 29.6 mJ/m², the saturated volume fraction of mechanical twins for this cross-section layer of the graded alloy was significantly lower versus the homogenous 0.4C alloy. This indicates that the SFE may not be the only parameter which determines the saturated twin volume fraction. Based on the EBSD phase maps observed in Figure 6-21, the dominant deformation product for this layer (250-500 microns from the surface) of the carbon graded alloy was determined to be mechanical twins.



(a) ε=0.15





Figure 6-21: EBSD phase maps from the mid-thickness cross-section (250-500 microns from the surface) of carbon graded alloy as a function of true strain.

Figure 6-22 displays the EBSD phase maps from the core cross-section (500-750 microns from the surface) of the carbon graded Fe-30Mn-C alloy as a function of applied strain. At $\varepsilon = 0.15$ (Figure 6-22(a)), the core microstructure of the steel comprised an equiaxed austenite matrix with some annealing twins. When the applied strain approached 0.2, mechanical twins started to appear within the core microstructure, as displayed in Figure 6-22(b). At high applied strains (Figure 6-22(c) and (d)), the mechanical twin

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

population increased significantly with applied strain. From Figure 6-20, it can be found that the twin volume fraction at higher strains for the core layer was significantly higher compared to the mid-thickness layer. This was attributed to the SFE profile of this steel (Figure 6-16), where SFE increased to 31.9-32.5 mJ/m² for the core layer. The saturated volume fraction of mechanical twins for the mid-thickness layer of the graded steel was similar to that observed for the homogenous 0.4C alloy with the SFE of 29.6 mJ/m² (Figure 6-6). Based on the EBSD phase maps observed in Figure 6-22, the dominant deformation product for the core cross-section layer of the carbon graded Fe-30Mn-C alloy was concluded to be mechanical twins.





50 µm

6.2.5 Fractography

50 µm

The fracture surface of the carbon graded steel was examined after uniaxial tensile testing to observe features presented in the damage zone and determine the main fracture mode. Figure 6-23 illustrates the SEM micrographs from the fracture surface of the alloy. Micro-cracks, micro-voids and ductile tearing features were observed with a small area fraction of cleavage facets being observed within the near surface cross-section (Figure 6-23(a)). This is consistent with the small area fraction of the relatively brittle ε martensite phase detected by EBSD within the near-surface cross-section microstructure of the steel (Figure 6-19). Thus, the dominant fracture mode was determined to be ductile for this alloy.



(a) near surface (0-250 μm from the surface)

(b) mid-thickness (250-500 µm from the



surface)

(c) core (500-750 μ m from the surface)

Figure 6-23: SEM micrographs from the fracture surface of the carbon graded Fe-30Mn-C alloy.

The sample cross-section near the fracture surface, sectioned along the tensile direction, of the carbon graded steel was also examined after uniaxial tensile testing to

Ph.D. Thesis – M. Ghasri Khouzani

McMaster – Materials Science & Engineering.

identify the main damage mechanism. Figure 6-24 displays typical SEM micrographs from the cross-section near the fracture surface of the graded Fe-30Mn-C alloy. In the near surface cross-section layer of the fracture sample, micro-voids and cracks were frequently observed along austenite/ɛ-martensite interfaces, as illustrated in Figure 6-24(a). Void nucleation at austenite/ε-martensite interfaces was found to be the dominant damage mechanism for the near surface layer of the graded alloy. This was consistent with other studies in the literature concerning ε -martensite formation alloys tensile tested to failure (Koyama et al., 2011, Lee et al., 2014, Liang, 2008, Yang, 2010). In the midthickness cross-section layer of the fracture sample (Figure 6-24(b)), significant occurrences of void nucleation at grain boundaries and decohesion of inclusions were observed. Thus, the damage mode for this cross-section layer of the graded alloy was concluded to be void nucleation at grain boundaries and inclusion/matrix interfaces. In the core cross-section of the alloy (Figure 6-24(c)), decohesion of a series of inclusions was observed, indicating that this was the dominant damage mechanism for this crosssection layer of the graded alloy. The critical damage mechanism for the global fracture of the carbon graded alloy was determined to be void nucleation at austenite/ɛ-martensite interfaces, which caused the near surface layer crack first and the effective cross-section to be reduced.





(a) near surface cross-section (0-250 μ m from



the surface)





(c) core cross-section (500-750 µm from the surface)

Figure 6-24: SEM micrographs from cross-section near fracture surface, sectioned along tensile direction, of carbon graded Fe-30Mn-C alloy.

6.2.6 Summary of Carbon Graded Fe-30Mn-C Alloy

The as-decarburized microstructure of the carbon graded alloy consisted of an austenite matrix and some annealing twins. Upon deformation, the microstructure of the surface layer (0-250 microns from the surface) was composed of austenite and mechanical ε -martensite with some deformation twins, where the volume fractions of ε -

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering. martensite and twins increased with increasing deformation. For the inner layers (250-750 microns from the surface), the deformed microstructures comprised a fully austenitic matrix and mechanical twins, where the twin volume fraction increased with strain level and also towards the core with the similar trend to that of the homogenous alloys. The saturated volume fraction of twins increased from the surface towards the core due to the increase in the SFE value.

The flow curve of the carbon graded alloy displayed serrations, which was attributed to the Portevin-LeChatlier (PLC) or dynamic strain aging (DSA) effect. The tensile properties of the carbon graded steel were not as good as the monolithic Fe-30Mn-0.6C steel because of their lower sustained high work hardening rates.

7 DISCUSSION

In this chapter, the mechanical behaviour and microstructural evolution findings for all experimental alloys will be discussed. Moreover, a general discussion on the effect of SFE as a critical physical property of each alloy will be provided. Finally, the work hardening behaviour of all homogenous alloys will be modelled using a modified Kocks-Mecking approach where the mechanical property evolution will be linked to the ε martensite and twinning kinetics observed for these alloys.

7.1 Comparison of Deformation Product Formation between Homogenous Fe-22Mn-C and Fe-30Mn-C Alloys

Two deformation products were detected in both the Fe-22Mn-C and Fe-30Mn-C alloy systems: mechanical twins and mechanical ε -martensite. In the present section, the formation of these products in both alloy systems will be discussed and compared.

7.1.1 Mechanical Twinning

Mechanical twins were found to be the dominant deformation products for the Fe-22Mn-0.4C, Fe-22Mn-0.6C, Fe-30Mn-0.4C and Fe-30Mn-0.6C alloys. Considering the twin volume fraction change with true strain for the Fe-22Mn-C alloys (Figure 5-8), the onset of twinning was found to be between $\varepsilon = 0.05$ and $\varepsilon = 0.1$ for both alloys. This is in agreement with similar studies on Fe-20Mn-1.2C and Fe-18Mn-0.6C-1.5Al TWIP steels (Renard and Jacques, 2012, Jin and Lee, 2009). The activation stress for twinning can be calculated using the inflection points associated with the increased d σ /d ε values in the work hardening curves (Figure 5-3(b)) to be $\varepsilon = 0.05$ ($\sigma = 300$ MPa) and $\varepsilon = 0.08$ ($\sigma =$ Ph.D. Thesis – M. Ghasri KhouzaniMcMaster – Materials Science & Engineering.400 MPa) for the 22Mn-0.4C and 0.6C alloys, respectively. From the tensile and workhardening curves of the Fe-20Mn-1.2C steel of Renard and Jacques (Renard and Jacques,2012), the corresponding stress for the activation of twin formation was determined to be

In the case of Fe-30Mn-C system, the onset of mechanical twinning was found to lie between $\varepsilon = 0.1$ and $\varepsilon = 0.2$ for both the 0.4C and 0.6C alloys (see Figure 6-6). From the inflection points in the d σ /d ε work hardening plots (Figure 6-3(b)), the onset strain for the mechanical twinning was determined to be $\varepsilon = 0.15$ ($\sigma = 570$ MPa) and $\varepsilon = 0.18$ ($\sigma = 660$ MPa) for the 0.4C and 0.6C alloys, respectively.

approximately 650 MPa.

For both alloy systems, the stress for the activation of mechanical twinning increased with increasing alloy SFE. This relationship is further explored in Figure 7-1, which presents the stress for mechanical twin activation (σ_t) as a function of SFE, supplemented by data from Renard and Jacques (Renard and Jacques, 2012), Yang (Yang, 2010) and Jin and Lee (Jin and Lee, 2009). It should be noted that all the SFE values in Figure 7-1 were calculated using the multicomponent SFE model of Dumay et al. (Dumay et al., 2008). Using the model form originally proposed by Byun (Byun, 2003), for the relationship between the activation stress for twinning and alloy SFE for 316L stainless steel, it was found that the data in Figure 7-1 obeyed the following linear relationship:

$$\sigma_t = (2.95 \pm 0.07) \frac{\Gamma_{SFE}}{b_p} \quad r^2 = 0.99 \quad \Gamma_{SFE} \ge 13.6 \text{ mJ/m}^2 \tag{7-1}$$

170

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

where σ_t is in Pa, Γ_{SFE} (J/m²) the stacking fault energy and b_p (m) the magnitude of the Burgers vector for a Shockley partial dislocation. Since the magnitude of the Burgers vector for a perfect dislocation in TWIP steels was previously reported to be 0.25 nm (Bouaziz et al., 2008), b_p was calculated to be 0.144 nm. It can be seen that the onset stress for mechanical twinning and the consequent increase in work hardening rate of high-Mn TWIP steels can be proportionally related to the alloy SFE through Eq. 7-1. However, it should be noted that Eq. 7-1 is only applicable to the case of high-Mn TWIP steels presented in Figure 7-1. For other high-Mn steels, the constant in Eq. 7-1 may be different as it differs from the value provided for 316L stainless steel (Byun, 2003).



Figure 7-1: Stress for the initiation of twinning as a function of SFE following the proposed model for of Byun (Byun, 2003), supplemented by the data of Renard and Jacques (Renard and Jacques, 2012), Yang (Yang, 2010) and Jin and Lee (Jin and Lee, 2009).

From Figure 5-8, it can be seen that the volume fraction of twins saturated for both the Fe-22Mn-0.4C and Fe-22Mn-0.6C alloys when true strain reached approximately 0.3 ($\sigma \approx 830$ MPa for the 0.4C alloy and $\sigma \approx 860$ MPa for the 0.6C alloy). This corresponded to a drop in the work hardening rate for these alloys (see Figure 5-3(b)), as would be consistent with a significant deceleration in the creation of new obstacles for dislocation motion. The twin saturation strain (or corresponding drop in work hardening rate) was reported to be $\varepsilon = 0.3$ for the Fe-20Mn-1.2C steel of Renard and Jacques

McMaster – Materials Science & Engineering.

(Renard and Jacques, 2012) and $\varepsilon = 0.37$ for the Fe-18Mn-0.6C-1.5Al alloy investigated by Jin and Lee (Jin and Lee, 2009). In the case of the present Fe-30Mn-0.4C and Fe-30Mn-0.6C alloys, however, a sharp increase in twin volume fraction was observed for the 0.2-0.3 true strain range, while the increase in twin volume fraction decreased significantly for further strains (Figure 6-6). This corresponded to a drop in the strain hardening plots of these alloys presented in Figure 6-3(b). The good correspondence between twin volume fraction evolution and work hardening rate in the present work and similar studies from the same research group (Lu et al., 2014, Yang, 2010, Yang et al., 2010) confirm the studies of other authors in the literature (Allain et al., 2004a, Allain et al., 2004b, Barbier et al., 2009, Bouaziz et al., 2008, Bouaziz and Guelton, 2001, Gutierrez-Urrutia and Raabe, 2012, Jin and Lee, 2009, Renard and Jacques), which attributed the high work hardening rates of high manganese TWIP steels to the role of mechanical twins in decreasing the mean free path for dislocation motion - i.e. the dynamic Hall-Petch effect. These are contrary to the findings of other authors, in which the high work hardening rates for high manganese TWIP steels were concluded to be the consequence of the PLC or DSA effect (Adler et al., 1986, Dastur and Leslie, 1981).

For both alloy systems, the saturated volume fraction of mechanical twins increased with increasing alloy carbon level. Furthermore, for the same alloy carbon level, the saturated twin volume fraction increased with increasing manganese content. Thus, it can be expected that the saturated population of mechanical twins increased with increasing alloy SFE. This is depicted in Figure 7-2. It can be seen that the saturated volume fraction of twins generally increased with increasing the SFE; however, the increase was not significant at high SFE values. This can be explained by suppression of mechanical twinning for high-Mn steels with relatively high SFE values (> 33 mJ/m^2) and the increasing dominance of dislocation glide as the primary deformation mechanism (Remy and Pineau, 1976).



Figure 7-2: Saturated twin volume fraction as a function of SFE for various TWIP alloys.

7.1.2 Mechanical Martensitic Transformation

Mechanical ε -martensite was found to be the dominant deformation product for the Fe-22Mn-0C and Fe-22Mn-0.2C alloys. A small volume fraction of mechanical ε martensite formed during the deformation of the Fe-22Mn-0.4C and Fe-30Mn-0C alloys. For the Fe-22Mn-C alloys, the largest increase in ε -martensite volume fraction was observed when the true strain increased from 0.02 to 0.1. Further deformation resulted in a significant decrease in the rate of ε -martensite formation (Figure 5-4). In the case of the Fe-30Mn-0C alloy, however, the ε -martensite volume fraction continued to increase until necking, although the curve saturated as the true strain reached 0.3 (Figure 6-4).

The change in saturated volume fraction of ε -martensite as a function of SFE for some high manganese steels of varying compositions is presented in Figure 7-3. To construct this plot, the experimental results in this work (Fe-22Mn-0C, Fe-22Mn-0.2C, Fe-22Mn-0.4C, Fe-22Mn-0.6C and Fe-30Mn-0C alloys) were used as well as the findings of some other authors taken from the literature (Lee et al., 2014, Liang et al., 2009, Yang et al., 2010). It is worth noting that Lee et al. (Lee et al., 2014) studied microstructural evolution of a Fe-12Mn-0.6C-0.06N alloy with tensile deformation at 293 K whereas Liang et al. (Liang et al., 2009) investigated the microstructural evolution of Fe-24.3Mn-0.02C and Fe-30.8Mn-0.02C alloys deformed via uniaxial tension at 77 K and 293 K. Moreover, the investigation of Yang et al. was on a series of Fe-21.5Mn alloys with carbon contents of 0.25, 0.40 and 0.56 wt. % deformed at 293 K (Yang et al., 2010). It should also be noted that the SFE of the Fe-12Mn-0.6C-0.06N alloy was that reported by Lee et al. (Lee et al., 2014), whereas the SFE of the other alloys was calculated using the SFE model of Dumay et al. (Dumay et al., 2008). As it can be seen in Figure 7-3, all the data were fitted by the same sigmoidal plot, indicating that the volume fraction of ε martensite at fracture was not a strong function of alloy chemical composition, but a combination of chemical composition and temperature determining the SFE value. It must be cautioned that this trend is only valid for the high-Mn steels plotted. When the SFE was less than approximately 6 mJ/m² (Figure 7-3), the saturated ε -martensite volume fraction did not change significantly with SFE and was constant at approximately 0.7.



Figure 7-3: Saturated ε -martensite volume fraction as a function of SFE for various high-Mn Fe-Mn-C alloys.

Further examination of Figure 7-3 will show that the saturated volume fraction of ε -martensite also became relatively independent of the SFE when the alloys SFE was greater than approximately 12 mJ/m², which can be explained by the change in the dominant deformation products from mechanical ε -martensite to mechanical twins. The role of ε -martensite as initiation site for fracture was previously observed by the cleavage characteristics of the fracture surface observed in Figure 5-10. Moreover, these findings

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

are consistent with the fracture analyses made in the literature (Koyama et al., 2011, Lee et al., 2014, Liang, 2008, Yang, 2010). Basically, similar to dual-phase steels, the mechanical property contrast between the soft austenite matrix and hard ε -martensite phase leads to separation of these two phases initiating from the phase boundary. For the Fe-22Mn-0C and Fe-22Mn-0.2C alloys, the large volume fraction of ε -martensite (Figure 5-4) resulted in the rapid decline in work hardening curve observed (Figure 5-3(b)) and also the relatively low uniform elongation for these alloys compared the other alloys with higher SFE values (Table 5-2).

7.1.3 Combination of Mechanical Twinning and Martensitic Transformation

In the case of alloys with a combination of mechanical ε -martensite and twins as deformation products – e.g. the Fe-22Mn-0.4C alloy with a SFE of 15.0 mJ/m² – the situation became more complex. Nevertheless, from Figure 5-4 and Figure 5-8, it can be seen that the sum of the saturated ε -martensite volume fraction ($f_{v,\varepsilon,0.4C} = 0.12$) and saturated twin volume fraction ($f_{v,twins,0.4C} = 0.25$) for the Fe-22Mn-0.4C alloy was very similar to that of the Fe-22Mn-0.6C alloy ($f_{v,total,0.6C} = 0.37$). Figure 7-4 depicts the sum of the volume fractions of ε -martensite and twins for the Fe-22Mn-0.4C, Fe-22Mn-0.6C, Fe-30Mn-0.4C and Fe-30Mn-0.6C alloys. Since no ε -martensite was observed within the deformed microstructures of the Fe-30Mn-0.4C and Fe-30Mn-0.6C alloys (Figure 6-8 and Figure 6-9), the ε -martensite volume fraction of these alloys was taken to be zero for all strains. For comparison purpose, the data for twin volume fraction at fracture from the work of Renard and Jacques (Renard and Jacques, 2012) and Lu et al. (Lu et al., 2014) were also added to Figure 7-4. It is worth noting that Renard and Jacques (Renard and

McMaster – Materials Science & Engineering.

Jacques, 2012) investigated the microstructural evolution with uniaxial tensile deformation of a Fe-19.9Mn-1.2C alloy whereas Lu et al. (Lu et al., 2014) studied the microstructural evolution of a Fe-22Mn-0.6C alloy deformed via uniaxial tension (UT) and plane strain tension (PST). The SFE of these alloys was calculated using SFE model of Dumay et al. (Dumay et al., 2008) to be approximately 30 mJ/m² and 21 mJ/m², respectively. For all alloys except the Fe-30Mn-0.6C alloy, it can be seen that the microstructure saturated with deformation products (Figure 7-4). In the case of Fe-30Mn-0.6C alloy, the twin volume fraction at fracture could not be determined due to the large amount of un-indexed areas in the EBSD phase map. However, since the plot slope for this alloy decreased significantly at higher strains, the alloy microstructure was expected to saturate at fracture. From Figure 7-4, it can also be seen that in all cases, fracture initiated when the sum of the volume fractions of ε -martensite and twins approached approximately 0.35 for all alloys, regardless of the alloy chemical composition or strain path. This indicates that the initiation of the critical damage for these high-Mn alloys was strongly dependent on the kinetics of the mechanical twinning and mechanical εmartensite formation. This conclusion is similar to that made for ε -martensite forming alloys, where the ε -martensite formation kinetics was found to play the main role in the critical damage of the alloys. For this reason, the kinetics of the mechanical twinning and mechanical ε -martensite formation was taken into account in the modelling of the work hardening rate of the experimental alloys, as presented in the next section.



Figure 7-4: Sum of ε -martensite and twin volume fractions versus true strain for the Fe-22Mn-0.4C, Fe-22Mn-0.6C, Fe-30Mn-0.4C and Fe-30Mn-0.6C alloys, including volume fraction of twins at fracture from Renard and Jacques (Renard and Jacques, 2012) and Lu et al., 2014).

7.2 Modelling the Work Hardening Rate of Homogenous Alloys

The Kocks-Mecking model (Kocks and Mecking, 2003) for the work hardening rate of a polycrystalline solid was reviewed in §2.4.1. The work hardening rate due to dislocation glide (Θ_{DG}) was defined as (Kocks and Mecking, 2003, Liang et al., 2009):

$$\Theta_{DG} = \Theta_0 (1 - \frac{1}{\mu} (\frac{\sigma_{V0}}{\mu_0})^{-1} (\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}})^{-C \frac{T}{T_M} \chi^n} \sigma)$$
(7-2)

where Θ_0 is the athermal work hardening rate, μ the shear modulus of the material, $\frac{\sigma_{V0}}{\mu_0}$ the ratio of scaling stress to shear modulus at 0K, $\dot{\varepsilon}$ the applied strain rate, *T* the test temperature, χ the material SFE, T_M the material melting point, σ the flow stress, and $\dot{\varepsilon}_0$, *C* and *n* are fitting parameters. The $\frac{\sigma_{V0}}{\mu_0}$ ratio is a function of alloy SFE, and can be found using Figure 2-11 (Kocks and Mecking, 2003).

Assuming that the contribution of phase transitions (twinning and ε -martensite formation in the present case) to the work hardening behaviour to be proportional to the volume fraction of the deformation product, Liang et al. (Liang, 2008, Liang et al., 2009) developed the following equation based on Remy's model (Remy, 1978) for the additional contribution of mechanical twinning to the net work hardening rate:

$$\Theta_{ph} = C'N\mu b \frac{1}{2t} f(\sigma)$$
(7-3)

where C' is a fitting parameter, N the average number of dislocations piling up at twin boundaries or γ/ϵ interfaces, μ the material shear modulus, b the magnitude of the Burgers vector, t the average thickness of the deformation products and f(σ) the volume fraction of deformation products as a function of flow stress. According to Remy's model (Remy, 1978), N was approximately 48 at 293 K.

The overall work hardening rate (Θ) is assumed to be the sum of the dislocation glide contribution (Eq. 7-2) and the phase transition contribution (Eq. 7-3), as follows:

$$\Theta = \Theta_0 \left(1 - \frac{1}{\mu} \left(\frac{\sigma_{V0}}{\mu_0}\right)^{-1} \left(\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}}\right)^{-C \frac{T}{T_M} \chi^n} \sigma\right) + C' N \,\mu b \frac{1}{2t} f(\sigma)$$
(7-4)

It is worth noting that for the TWIP/TRIP alloys (e.g. Fe-22Mn-0.4C steel) the phase transition contribution (the second term in Eq. 7-4) comprises both twinning and ε -martensitic transformation. For all experimental alloys in this work except the Fe-22Mn-0C alloy, the following general form sigmoidal equation was found to fit f(σ):

$$f(\sigma) = \frac{A_{1}}{1 + \exp[A_{2}(A_{3} - \sigma)]}$$
(7-5)

where A1, A2 and A3 are fitting parameters. In the case of the Fe-22Mn-OC alloy, the volume fraction of ε -martensite as a function of stress was fitted by the following sigmoidal equation:

$$f_{22Mn-0C}^{\varepsilon}(\sigma) = 0.525 + \frac{0.242}{1+10^{0.0128(588.35-\sigma)}}$$
(7-6)

Numerical values of the parameters used in modelling the work hardening behaviour of all homogenous alloys are listed in Table 7-1, Table 7-2 and Table 7-3 at the end of the section.

7.2.1 ε-Martensite Formation Alloys

As was mentioned in Chapter 5, the dominant deformation products for the Fe-22Mn-0C and Fe-22Mn-0.2C alloys were found to be mechanical ε -martensite. The evolution of ε -martensite volume fraction with true stress for these alloys is presented in Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

Figure 7-5. It can be seen that the volume fraction of ε -martensite as a function of true stress for both the alloys were well represented using a sigmoidal curve. However, the form of the sigmoidal function for the alloys was not the same, being Eq. 7-5 for the Fe-22Mn-0.2C alloy (see Table 7-2) and Eq. 7-6 for the Fe-22Mn-0C alloy. These expressions were used to calculate the martensitic transformation contribution to the work hardening of both the alloys per Eq. 7-4.



Figure 7-5: Fitting plots of ε -martensite volume fraction versus true stress for the Fe-22Mn-0C and Fe-22Mn-0.2C alloys.

The comparison between the experimental work hardening results and the calculated work hardening rates for the ε -martensite formation Fe-22Mn-OC and Fe-

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

22Mn-0.2C alloys is presented in Figure 7-6. For both alloys, it can be seen that the model agrees with the experimental strain hardening behaviour. In the case of dislocation glide contribution, the work hardening rate decreased continuously during deformation as dynamic recovery was the primary contributor to dislocation glide work hardening (Eq. 7-2) (Kocks and Mecking, 2003). The $\gamma \rightarrow \varepsilon$ martensitic transformation contribution to the work hardening, however, sigmoidally increased with increasing stress, per the experimental transformation kinetics, and contributed positively to the work hardening rate through the creation of new obstacles until ε -martensite saturation was reached (Figure 7-5). Following ε -martensite saturation at approximately $\sigma = 700$ MPa for the Fe-22Mn-0C alloy and at $\sigma = 650$ MPa for the Fe-22Mn-0.2C alloy, the phase transition no longer contributed significantly to the work hardening rate and the work hardening rate was primarily a function of the dynamic recovery term.



(b) Fe-22Mn-0.2C alloy

Figure 7-6: Comparison between the work hardening experimental results and model (Eq. 7-4) for the Fe-22Mn-0C and Fe-22Mn-0.2C alloys.

7.2.2 Twin Formation Alloys

Mechanical twins were found to be the only deformation product for the Fe-22Mn-0.6C, Fe-30Mn-0.2C, Fe-30Mn-0.4C and Fe-30Mn-0.6C alloys. Figure 7-7 depicts the evolution of twin volume fraction as a function of true stress for these alloys. It can be seen that the experimental curves for all the alloys were fitted by sigmoidal plots of the form shown in Eq. 7-5, where the detailed parameters can be found in Table 7-2 and Table 7-3 for the 22Mn and 30Mn alloys, respectively. This equation was used to calculate the contribution of the mechanical twinning to the overall work hardening rate of the alloys through the modified Kocks-Mecking model specified in Eq. 7-4.



Figure 7-7: Fitted plots of twin volume fraction evolution with true stress for the twin formation alloys; symbol lines denote experimental results and solid lines are fitted curves.

The experimental results and model (Eq. 7-4) for the work hardening behaviour of the Fe-22Mn-0.6C and Fe-30Mn-0.2C alloys are presented in Figure 7-8. In both cases, the model was in agreement with the experimental results. For the Fe-22Mn-0.6C alloy (Figure 7-8(a)), the mechanical twinning contribution to the overall work hardening rate became dominant from $\sigma \approx 420$ MPa, leading to increase in work hardening rate by the dynamic Hall-Petch effect (Allain et al., 2004a, Bouaziz and Guelton, 2001, Shiekhelsouk et al., 2009). When the true stress approached $\Rightarrow 0$ MPa, the increase in work

hardening rate ceased as a consequence of mechanical twin saturation, as observed in Figure 7-7. Thus, the dislocation glide contribution to the overall work hardening became dominant until fracture, resulting in continuous decrease in work hardening rate due to dynamic recovery (Kocks and Mecking, 2003).

In the case of the Fe-30Mn-0.2C alloy (Figure 7-8(b)), the dislocation glide contribution to the work hardening rate seemed to be dominant during the entire deformation of the alloy, leading to a continuous decrease to work hardening rate until fracture. However, for true stress values of 400-700 MPa, the overall work hardening curve decrease became less steep (Figure 7-8(b)) as a result of mechanical twinning. Nevertheless, the mechanical twinning contribution was not high enough to lead to an increase in the work hardening rate. From the physical viewpoint, the volume fraction of mechanical twins was not high enough (see Figure 7-7) to significantly reduce the mean free paths for dislocation motion and prevent dynamic recovery.



Figure 7-8: Comparison between the work hardening experimental results and model (Eq. 7-4) for the Fe-22Mn-0.6C and Fe-30Mn-0.2C alloys.

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

Figure 7-9 depicts the comparison between the work hardening experimental results and modified Kocks-Mecking model for the Fe-30Mn-0.4C and Fe-30Mn-0.6C TWIP steels. In the case of the Fe-30Mn-0.4C alloy (Figure 7-9(a)), the model work hardening rate decreased continuously from the onset of plastic deformation to ∞ 500 MPa. This was attributed to the dominance of the dislocation glide contribution to the work hardening rate. When true stress approached $\sigma \approx$ 500 MPa, the work hardening rate started to increase slightly due to the mechanical twinning contribution. However, the increase in work hardening rate stopped at \approx 920 MPa because mechanical twin formation saturated, as can be seen in Figure 7-7. After reaching a true stress of $\sigma \approx$ 920 MPa, the dislocation glide contribution became dominant again, resulting in a decrease in the overall work hardening rate due to the dominance of dynamic recovery until fracture.

The work hardening model for the Fe-30Mn-0.6C alloy (Figure 7-9(b)) was similar to that of the Fe-30Mn-0.4C alloy (Figure 7-9(a)). However, the dominance of the mechanical twinning contribution started at a higher stress ($\sigma \approx 620$ MPa) and also ended at a higher stress ($\sigma \approx 1050$ MPa) compared to the Fe -30Mn-0.4C alloy. This was attributed to the higher SFE of the Fe-30Mn-0.6C alloy versus that of the Fe-30Mn-0.4C alloy, as was discussed earlier. Moreover, the mechanical twinning for the Fe-30Mn-0.6C alloy led to a higher increase in the work hardening rate, which is explained by considering the higher volume fraction of mechanical twins observed for this alloy (see Figure 7-7).



Figure 7-9: Comparison between the work hardening experimental results and model (Eq. 7-4) for the Fe-30Mn-0.4C and Fe-30Mn-0.6C alloys.

7.2.3 Mixed Deformation Product Alloys

The deformation products for the Fe-22Mn-0.4C and Fe-30Mn-0C alloys were a combination of mechanical ε-martensite and mechanical twins. Each of these products can contribute separately to the overall work hardening of the alloys. In other words, two phase transition terms for work hardening (Θ_{ph}) in Eq. 7-3, should be calculated; one for mechanical twinning and one for mechanical ε -martensite formation. The evolution of the ε-martensite and twin volume fraction with true stress for the Fe-22Mn-0.4C and Fe-30Mn-0C alloys is presented in Figure 7-10. From Table 7-2 and Table 7-3, it can be seen that all the experimental plots were fitted using sigmoidal curves of the general form presented in Eq. 7-5. For the Fe-22Mn-0.4C alloy, the saturated volume fraction of twins was two times greater than that of ε -martensite, as shown in Figure 7-10. Thus, mechanical twinning was expected to display a more significant contribution to the overall work hardening of this alloy. In the case of the Fe-30Mn-0C alloy, however, the saturated volume fraction of both ε -martensite and twins was approximately the same z(0.12), as can be seen in Figure 7-10. Thus, the similar contribution of these deformation products to the overall work hardening of the Fe-30Mn-0C alloy was expected.



(a) ε-martensite volume fraction as a function of true stress



(b) twin volume fraction as a function of true stress

Figure 7-10: Fitted plots of the deformation product evolution with true stress in the Fe-22Mn-0.4C and Fe-30Mn-0C alloys.
Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

The work hardening experimental results and model (Eq. 7-4) for the Fe-22Mn-0.4C and Fe-30Mn-0C alloys are presented in Figure 7-11. For the Fe-22Mn-0.4C alloy (Figure 7-11(a)), the overall work hardening rate initially increased until $\sigma \approx 400$ MPa due to ε -martensite formation. This stage was relatively short because the ε -martensite formation saturated quickly (see Figure 7-10(a)). For the true stress range of 400-800 MPa, the mechanical twinning contribution to the overall work hardening rate was dominant, leading to an increase in the overall work hardening rate. When the true stress approached 800 MPa, the increase in work hardening rate stopped owing to mechanical twinning saturation, as shown in Figure 7-10(b). From this point, the dislocation glide contribution became dominant, resulting in the continuous decrease in the work hardening rate until fracture as dynamic recovery of dislocation cells dominated.

In the case of the Fe-30Mn-OC alloy, the dislocation glide contribution to the overall work hardening was dominant during the entire deformation, leading to the observed continuous decrease in the work hardening rate as a consequence of dynamic recovery (Kocks and Mecking, 2003). However, the rate of decrease for the overall work hardening rate became lower when the true stress reached approximately 300 MPa. This was attributed to the contribution of mechanical twinning and ε -martensite formation. However, this contribution was too small to stop the decrease in the overall work hardening rate. In other words, the volume fraction of the ε -martensite and mechanical twins was not large enough to result in significant enough dislocation blocking through reducing the mean free paths for dislocation motion (see Figure 7-10).



Figure 7-11: Comparison between the work hardening experimental results and model (Eq. 7-4) for the Fe-22Mn-0.4C and Fe-30Mn-0C alloys.

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

From the above mentioned modelling work, it can be concluded the work hardening behaviour of the high manganese steels, with various compositions and deformation products can be described using a modified Kocks-Mecking formalism comprising a combination of the conventional dislocation glide term – which has been well established in the literature (Kocks and Mecking, 2003, Liang et al., 2009) – and measured transformation (ϵ -martensite formation and/or mechanical twinning) kinetics for the alloys. The advantage of the model is that it correlates the mechanical behaviour to the microstructural evolution – the evolution of the deformation products – for the alloys. Table 7-1. Numerical values of parameters used for modelling the work hardening of all homogenous Fe-22Mn-C alloys.

Parameter	Numerical Value	Reference
Т	293 (K)	
μ	65 (GPa)	(Bouaziz et al., 2008)
$\dot{\mathcal{E}}_0$	10^{5}	(Liang, 2008)
Ė	$6.67 \times 10^{-4} (s^{-1})$	
n	0.105	(Liang, 2008)
b	0.25 (nm)	(Bouaziz et al., 2008)
t	300 (nm)	(Remy, 1978)

Table 7-2. Numerical values of parameters used for modelling the work hardening rate as a function of various homogenous Fe-22Mn-C alloys.

Parameter	0C alloy	0.2C alloy	0.4C alloy	0.6C alloy	Reference
Θ_0	5569 (MPa)	3090 (MPa)	1829 (MPa)	2069 (MPa)	
$(\frac{\sigma_{_{V0}}}{\mu_0})^{0.5}$	0.190	0.175	0.167	0.161	(Kocks and Mecking, 2003)
T_M	1705 (K)	1698 (K)	1685 (K)	1676 (K)	Thermo-Calc
С	-0.424	-0.149	-0.094	-0.047	
χ	0.1 (mJ/m ²)	5.5 (mJ/m ²)	15.0 (mJ/m ²)	19.7 (mJ/m ²)	(Dumay et al., 2008)
C'	1.4	1.5	3.8 for twinning and 3.5 for martensitic transformation	4.0	
A_1		0.6313	0.2513 for twinning and 0.1204 for martensitic transformation	0.3434	
A_2		0.0071	0.0071 for twinning and 0.0171 for martensitic transformation	0.0081	
A_3		336.04	595.29 for twinning and 284.80 for martensitic transformation	595.79	

Table 7-3. Numerical values for parameters used for modelling the work hardening rate as a function of various homogenous Fe-30Mn-C alloys.

Parameter	0C alloy	0.2C alloy	0.4C alloy	0.6C alloy	Reference
Θ_0	2530 (MPa)	3013	3264	3168	
		(MPa)	(MPa)	(MPa)	
$(rac{\sigma_{_{V0}}}{\mu_{_0}})^{0.5}$	0.161				(Kocks and
		0.158	0.155	0.153	Mecking,
					2003)
T_M	1672 (K)	1663 (K)	1651 (K)	1641 (K)	Thermo-Calc
С	-0.171	-0.128	-0.061	-0.015	
χ	17.9 (mJ/m ²)	23.5	29.6	33.2	(Dumay et
		(mJ/m^2)	(mJ/m^2)	(mJ/m^2)	al., 2008)
C'	1.9 for both twinning and	2.5	2.4	2.1	
	martensitic transformation				
A_1	0.1882 for twinning and	0.2369	0.3806	0.4458	
	0.1527 for martensitic				
	transformation				
A_2	0.0062 for twinning and				
	0.0060 for martensitic	0.0071	0.0084	0.0084	
	transformation				
A ₃	565.36 for twinning and				
	494.54 for martensitic	336.04	732.64	768.68	
	transformation				

7.3 Comparison between the Mechanical Properties of the Homogenous and Carbon Graded Alloys

For both the Fe-22Mn and Fe-30Mn alloy systems, it was found that the mechanical properties of the carbon graded alloys were not as good as the parent homogenous Fe-22Mn-0.6C and Fe-30Mn-0.6C alloys. In this section, the difference between the mechanical properties of the carbon graded alloys and the parent alloys (Fe-22Mn-0.6C and Fe-30Mn-0.6C alloys) will be explained. From the macroscopic viewpoint, the carbon graded alloys can be considered to be laminar composites of the

Ph.D. Thesis – M. Ghasri KhouzaniMcMaster – Materials Science & Engineering.homogenous alloys with different carbon contents, as schematically illustrated in Figure

7-12. This can be understood from the carbon concentration profiles of the carbon graded alloys presented in Figure 5-14 and Figure 6-14.



Figure 7-12: Schematic illustration of the carbon graded alloys as laminar composite. Assuming symmetry, only the surface to the core is illustrated.

As was observed in Chapter 5 and Chapter 6, the 0.6C alloy displayed the highest tensile strength and uniform elongation among the homogenous alloys in the both alloy systems (Figure 5-3(a), Table 5-2, Figure 6-3(a) and Table 6-2). Thus, the surface and mid-thickness layers of the carbon graded alloys (Figure 7-12) were expected to have lower tensile strengths and uniform elongations versus the corresponding layers in the homogenous 0.6C alloys. This resulted in the carbon graded alloys exhibiting a lower overall tensile strength and uniform elongation versus the 0.6C alloys. It should be noted that in the case of classic carbon graded steels, decarburization is used to produce a composite with a soft surface layer such as ferrite and a harder core layer such as

Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering. martensite (Embury and Bouaziz, 2010). In this case, the surface layer has a lower tensile strength but larger uniform elongation versus the core layer, leading to the carbon graded steel exhibiting optimum mechanical properties.

As was mentioned above, for both the Fe-22Mn and Fe-30Mn alloy systems, the carbon graded steels exhibited lower uniform elongation versus the parent Fe-22Mn-0.6C and Fe-30Mn-0.6C alloys. This can be explained by considering the fracture origin of the carbon graded alloys. As was observed in Chapter 5 and Chapter 6, a significant volume fraction of mechanical ε -martensite formed within the near surface layer of the carbon graded steels as a consequence of the plastic deformation. Since austenite/ɛ-martensite interfaces act as the nucleation sites for the voids (Koyama et al., 2011, Lee et al., 2014, Liang, 2008, Yang, 2010), the near surface layer of the carbon graded alloys cracked first, similar to the *\varepsilon*-martensite formation 22Mn-0C and 22Mn-0.2C alloys. Thus, the effective cross-section reduced and could not support the applied load, leading to early fracture (at lower strains versus the parent 0.6C alloys). This discussion can also be used to explain the lower uniform elongation of the carbon graded G3 alloy compared to the G1 and G2 alloys. In this case, the near surface layer in which mechanical ε-martensite was the dominant deformation products was thicker for the G3 alloy versus the G1 and G2 alloys. Since this layer cracks first, the effective cross-section for the G3 alloy was lower than the G1 and G2 alloys. Thus, the overall fracture for the G3 alloy occurred at lower strains.

8 CONCLUSIONS

8.1 Homogenous Fe-22Mn-C Alloys

The as heat-treated microstructure of the 0C and 0.2C alloys comprised austenite and thermal ε -martensite, with the dominant deformation products being mechanical ε martensite for these alloys. These deformation products resulted in the alloys exhibiting a high initial work hardening rate but low elongation compared to the parent 0.6C alloy. For the 0.4C alloy, the as heat-treated microstructure was fully austenitic, with the deformation products being significant volume fractions of mechanical twins in addition to a relatively small volume fractions of ε -martensite. The work hardening behaviour of this alloy resembled that of a typical TWIP steel, though lower sustained high work hardening rates versus the 0.6C alloy were observed for the 0.4C alloy. The as heattreated microstructure of the 0.6C alloy was fully austenitic, where the dominant deformation products were mechanical twins. These deformation products resulted in the high sustained work hardening rates observed for this alloy. As a consequence, the 0.6C alloy exhibited the highest UTS and uniform elongation among the homogenous Fe-22Mn-C alloys.

Both the mechanical ε -martensite and twin formation were found to follow a sigmoidal kinetic with strain. In the case of twin formation 0.4C and 0.6C alloys, the saturation volume fraction of twins was directly proportional to the alloy SFE. For ε -martensite formation alloys, the ε -martensite volume fraction at fracture was found to be strongly dependent on alloy SFE, where it declined sigmoidally with increasing alloy

Ph.D. Thesis – M. Ghasri KhouzaniMcMaster – Materials Science & Engineering.SFE. It was also found that the ε -martensite volume fraction at fracture – approximately0.7 – was independent of SFE for SFE $\leq 6 \text{ mJ/m}^2$. This indicated that the critical damagemechanism was determined by the kinetics of the ε -martensite formation, which was inturned dictated by the alloy SFE.

A modified Kocks-Mecking model was used for modelling the work hardening behaviour of all the 22Mn alloys. According to the model, the work hardening was the sum of the dislocation glide contribution and the phase transition contribution – mechanical twinning and/or mechanical ε -martensite formation – as dictated by the formation kinetics of both deformation products.

8.2 Carbon Graded Fe-22Mn-C Alloys

For all the carbon graded Fe-22Mn-C steels, the as-decarburized microstructure near the outer surface comprised a mixture of austenite and thermal ε -martensite, where the ε -martensite volume fraction decreased towards the core. The core microstructure for all steels was fully austenitic with some annealing twins. For all the alloys, the dominant deformation products changed from mechanical ε -martensite at the near-surface layer to a mixture of mechanical twins and ε -martensite or mechanical twins only at the inner crosssection layers. The yield stress of the G3 alloy was lower than that of G1 and G2 alloys due to its lower overall carbon content. This steel also displayed a higher initial work hardening rate than the other steels but a lower uniform elongation.

8.3 Homogenous Fe-30Mn-C Alloys

The as heat-treated microstructure of all the alloys comprised equiaxed austenite grains with some annealing twins. The deformation products for the 0C alloy consisted of small volume fractions of both mechanical ε -martensite and twins, leading to an alloy exhibiting a low work hardening rate typical of softer FCC metals. The deformation products for the 0.2C alloy were revealed to be small volume fractions of mechanical twins, with dislocation glide being the dominant deformation mode for this alloy. The deformed microstructures of both the 0.4C and 0.6C alloys revealed significant volume fractions of mechanical twins as deformation products, leading to alloys displaying high sustained work hardening rates.

The yield stress and tensile strength of the homogenous Fe-30Mn-C alloys increased with increasing alloy carbon content whereas the uniform elongation was not significantly affected.

The work hardening rate of all the alloys was successfully modelled using a modified Kocks-Mecking model, which was a combination of conventional dislocation glide term and measured transition (ε-martensite formation and/or mechanical twinning) kinetics for the alloys.

8.4 Carbon Graded Fe-30Mn-C Alloy

The as-decarburized microstructure of the carbon graded steel was composed of an equiaxed austenitic matrix with some annealing twins. Upon deformation, the microstructure of the surface layer (250 microns from the surface) consisted of austenite Ph.D. Thesis – M. Ghasri Khouzani McMaster – Materials Science & Engineering.

and mechanical ε -martensite with some deformation twins, where the volume fractions of ε -martensite and twins increased with strain level. For inner layers (250-750 microns from the surface), the deformed microstructures were fully austenitic with deformation twins, where the twin population increased with deformation level and also towards the core. The tensile properties of the carbon graded Fe-30Mn-C alloy were not as good as the monolithic Fe-30Mn-0.6C steel due to their lower sustained high work hardening rates.

REFERENCES

- Abbasi, A., Dick, A., Hickel, T. and Neugebauer, J. (2011) 'First-principles investigation of the effect of carbon on the stacking fault energy of Fe–C alloys', *Acta Materialia*, 59(8), pp. 3041-3048.
- Abel, A. and Muir, H. (1973) 'The Bauschinger effect and stacking fault energy', *Philosophical Magazine*, 27(3), pp. 585-594.
- Adeyev, V. and Petrov, Y. N. (1979) 'Stacking-fault energy of austenite in Fe-30-percent Ni-C alloys', *Fizika Metallov i Metallovedenie*, 48(6), pp. 1271-1275.
- Adler, P.H., Olson, G.B. and Owen, W.S. (1986) 'Strain hardening of Hadfield manganese steel', *Metallurgical and Materials Transactions A*, 17(10), pp. 1725-1737.
- Allain, S., Chateau, J.-P. and Bouaziz, O. (2002) 'Constitutive model of the TWIP effect in a polycrystalline high manganese content austenitic steel', *Steel research*, 73(6-7), pp. 299-302.
- Allain, S., Chateau, J.-P. and Bouaziz, O. (2004a) 'A physical model of the twinninginduced plasticity effect in a high manganese austenitic steel', *Materials Science and Engineering: A*, 387-389, pp. 143-147.
- Allain, S., Chateau, J.-P., Bouaziz, O., Migot, S. and Guelton, N. (2004b) 'Correlations between the calculated stacking fault energy and the plasticity mechanisms in Fe– Mn–C alloys', *Materials Science and Engineering: A*, 387-389, pp. 158-162.

- Allain, S., Chateau, J.-P., Dahmoun, D. and Bouaziz, O. (2004c) 'Modeling of mechanical twinning in a high manganese content austenitic steel', *Materials Science and Engineering: A*, 387-389, pp. 272-276.
- ASTM Standard. (2004) 'E8, Standard Test Methods for Tension Testing of Metallic Materials', Annual book of ASTM standards, 3, pp. 57-72.
- Barbier, D., Gey, N., Allain, S., Bozzolo, N. and Humbert, M. (2009) 'Analysis of the tensile behavior of a TWIP steel based on the texture and microstructure evolutions', *Materials Science and Engineering: A*, 500 (1-2), pp. 196-206.
- Bate, P.S. and Wilson, D.V. (1986) 'Analysis of the Bauschinger effect', Acta Metallurgica, 34(6), pp. 1097-1105.
- Bleck, W. and Phiu-On, K. (2005) 'Microalloying of cold-formable multi phase steel grades', *Materials Science Forum*, 500-501, pp. 97-114.
- Bouaziz, O., Allain, S. and Scott, C. (2008) 'Effect of grain and twin boundaries on the hardening mechanisms of twinning-induced plasticity steels', *Scripta Materialia*, 58(6), pp. 484-487.
- Bouaziz, O., Allain, S., Scott, C.P., Cugy, P. and Barbier, D. (2011a) 'High manganese austenitic twinning induced plasticity steels: a review of the microstructure properties relationships', *Current Opinion in Solid State and Materials Science*, 15(4), pp. 141-168.
- Bouaziz, O. and Guelton, N. (2001) 'Modelling of TWIP effect on work-hardening', *Materials Science and Engineering: A*, 319-321, pp. 246-249.

- Bouaziz, O., Zurob, H., Chehab, B., Embury, J.D., Allain, S. and Huang, M. (2011b)
 'Effect of chemical composition on work hardening of Fe-Mn-C TWIP steels', *Materials Science and Technology*, 27(3), pp. 707-709.
- Bracke, L., Mertens, G., Penning, J., De Cooman, B.C., Liebeherr, M. and Akdut, N. (2006) 'Influence of phase transformations on the mechanical properties of highstrength austenitic Fe-Mn-Cr steel', *Metallurgical and Materials Transactions A*, 37(2), pp. 307-317.
- Brofman, P.J. and Ansell, G.S. (1978) 'On the effect of carbon on the stacking fault energy of austenitic stainless steels', *Metallurgical Transactions A*, 9(6), pp. 879-880.
- Byun, T.S. (2003) 'On the stress dependence of partial dislocation separation and deformation microstructure in austenitic stainless steels', *Acta Materialia*, 51(11), pp. 3063-3071.
- Callister, W. D. and Rethwisch, D.G. (2012) Fundamentals of Materials Science and Engineering: An Integrated Approach. John Wiley & Sons.
- Chen, L., Kim, H.-S., Kim, S.-K. and De Cooman, B.C. (2007) 'Localized deformation due to Portevin-LeChatelier effect in 18Mn-0.6C TWIP austenitic steel', *ISIJ International*, 47(12), pp. 1804-1812.
- Christian, J. W. (2002) *The Theory of Transformations in Metals and Alloys (Part I+ II)*. Pregamon.

- Cotterill, R.M.J. and Doyama, M. (1966) 'Energy and atomic configuration of complete and dissociated dislocations. I. Edge dislocation in an fcc metal', *Physical Review*, 145(2), pp. 465-478.
- Dastur, Y.N. and Leslie, W.C. (1981) 'Mechanism of work hardening in Hadfield manganese steel', *Metallurgical Transactions A*, 12(5), pp. 749-759.
- De Cooman, B.C., Chen, L., Kim, H.S., Estrin, Y., Kim, S.K. and Voswinckel, H. (2009) 'State-of-the-science of high manganese TWIP steels for automotive applications', *Microstructure and Texture in Steels*, pp. 165-183.
- Dumay, A., Chateau, J.-P., Allain, S., Migot, S. and Bouaziz, O. (2008) 'Influence of addition elements on the stacking-fault energy and mechanical properties of an austenitic Fe–Mn–C steel', *Materials Science and Engineering: A*, 483-484, pp. 184-187.
- Embury, D. and Bouaziz, O. (2010) 'Steel-based composites: driving forces and classifications', *Annual Review of Materials Research*, 40, pp. 213-241.
- Ferreira, P.J. and Müllner, P. (1998) 'A thermodynamic model for the stacking-fault energy', *Acta Materialia*, 46(13), pp. 4479-4484.
- Frommeyer, G., Brüx, U. and Neumann, P. (2003) 'Supra-ductile and high-strength manganese-TRIP/TWIP steels for high energy absorption purposes', *ISIJ International*, 43(3), pp. 438-446.
- Fujita, H. and Ueda, S. (1972) 'Stacking faults and fcc (γ) hcp (ϵ) transformation in 18/8-type stainless steel', *Acta Metallurgica*, 20(5), pp. 759-767.

- Gebhardt, T., Music, D., Hallstedt, B., Ekholm, M., Abrikosov, I.A., Vitos, L. and Schneider, J.M. (2010) 'Ab initio lattice stability of fcc and hcp Fe–Mn random alloys', *Journal of Physics: Condensed Matter*, 22(29), pp. 295402.
- Gholizadeh, H., Draxl, C. and Puschnig, P. (2013) 'The influence of interstitial carbon on the γ-surface in austenite', *Acta Materialia*, 61(1), pp. 341-349.
- Grässel, O., Krüger, L., Frommeyer, G. and Meyer, L.W. (2000) 'High strength Fe–Mn– (Al, Si) TRIP/TWIP steels development—properties—application', *International Journal of Plasticity*, 16(10-11), pp. 1391-1409.
- Gutierrez-Urrutia, I. and Raabe, D. (2011) 'Dislocation and twin substructure evolution during strain hardening of an Fe–22wt.% Mn–0.6 wt.% C TWIP steel observed by electron channeling contrast imaging', *Acta Materialia*, 59(16), pp. 6449-6462.
- Gutierrez-Urrutia, I. and Raabe, D. (2012) 'Grain size effect on strain hardening in twinning-induced plasticity steels', *Scripta Materialia*, 66(12), pp. 992-996.
- Gutierrez-Urrutia, I., Zaefferer, S. and Raabe, D. (2010) 'The effect of grain size and grain orientation on deformation twinning in a Fe–22wt.% Mn–0.6 wt.% C TWIP steel', *Materials Science and Engineering: A*, 527(15), pp. 3552-3560.
- Hamada, A.S., Karjalainen, L.P. and Somani, M.C. (2007) 'The influence of aluminum on hot deformation behavior and tensile properties of high-Mn TWIP steels', *Materials Science and Engineering: A*, 467(1-2), pp. 114-124.
- Hirth, J.P. and Cohen, M. (1970) 'On the strength-differential phenomenon in hardened steel', *Metallurgical Transactions*, 1(1), pp. 3-8.

- Hulka, K. 'The role of niobium in cold rolled TRIP steel'. *Materials Science Forum*, 473-474, pp. 91-102.
- Idrissi, H., Renard, K., Ryelandt, L., Schryvers, D. and Jacques, P.J. (2010) 'On the mechanism of twin formation in Fe–Mn–C TWIP steels', *Acta Materialia*, 58(7), pp. 2464-2476.
- Jiang, B., Qi, X., Yang, S., Zhou, W. and Hsu, T.Y. (1998) 'Effect of stacking fault probability on γ–ε martensitic transformation and shape memory effect in Fe–Mn– Si based alloys', *Acta Materialia*, 46(2), pp. 501-510.
- Jin, J.-E. and Lee, Y.-K. (2009) 'Strain hardening behavior of a Fe–18Mn–0.6C–1.5Al TWIP steel', *Materials Science and Engineering: A*, 527(1-2), pp. 157-161.
- Karaman, I., Sehitoglu, H., Chumlyakov, Y.I., Maier, H.J. and Kireeva, I.V. (2001) 'The effect of twinning and slip on the Bauschinger effect of Hadfield steel single crystals', *Metallurgical and Materials Transactions A*, 32(13), pp. 695-706.
- Kelly, A., Groves, G.W. and Kidd, P. (2000) Crystallography and Crystal Defects. John Wiley & Sons.
- Khachaturyan, A.G. (2008) Theory of Structural Transformations in Solids. John Wiley & Sons.
- Kim, J., Estrin, Y. and De Cooman, B.C. (2013) 'Application of a dislocation densitybased constitutive model to Al-alloyed TWIP steel', *Metallurgical and Materials Transactions A*, 44(9), pp. 4168-4182.

- Kim, J., Lee, S.-J. and De Cooman, B.C. (2011) 'Effect of Al on the stacking fault energy of Fe–18Mn–0.6C twinning-induced plasticity', *Scripta Materialia*, 65(4), pp. 363-366.
- Kim, Y.-M., Shin, Y.-H. and Lee, B.-J. (2009) 'Modified embedded-atom method interatomic potentials for pure Mn and the Fe–Mn system', *Acta Materialia*, 57(2), pp. 474-482.
- Kocks, U.F. and Mecking, H. (2003) 'Physics and phenomenology of strain hardening: the FCC case', *Progress in Materials Science*, 48(3), pp. 171-273.
- Koyama, M., Sawaguchi, T., Lee, T., Lee, C.S. and Tsuzaki, K. (2011) 'Work hardening associated with ε-martensitic transformation, deformation twinning and dynamic strain aging in Fe–17Mn–0.6C and Fe–17Mn–0.8C TWIP steels', *Materials Science and Engineering: A*, 528(24), pp. 7310-7316.
- Lee, S., Estrin, Y. and De Cooman, B.C. (2014) 'Effect of the strain rate on the TRIP– TWIP transition in austenitic Fe-12 pct Mn-0.6 pct C TWIP steel', *Metallurgical and Materials Transactions A*, 45(2), pp. 717-730.
- Lee, T.-H., Oh, C.-S., Kim, S.-J. and Takaki, S. (2007) 'Deformation twinning in highnitrogen austenitic stainless steel', *Acta Materialia*, 55(11), pp. 3649-3662.
- Lee, Y.-K. (2012) 'Microstructural evolution during plastic deformation of twinninginduced plasticity steels', *Scripta Materialia*, 66(12), pp. 1002-1006.
- Lee, Y.-K. and Choi, C. (2000) 'Driving force for-γε martensitic transformation and stacking fault energy of γ in Fe-Mn binary system', *Metallurgical and Materials Transactions A*, 31(2), pp. 355-360.

- Li, K.P., Carden, W.P. and Wagoner, R.H. (2002) 'Simulation of springback', International Journal of Mechanical Sciences, 44(1), pp. 103-122.
- Liang, X. (2008) Structure and Mechanical Properties of Fe-Mn Alloys. Master Thesis, McMaster University.
- Liang, X., McDermid, J.R., Bouaziz, O., Wang, X., Embury, J.D. and Zurob, H.S. (2009) 'Microstructural evolution and strain hardening of Fe–24Mn and Fe–30Mn alloys during tensile deformation', *Acta Materialia*, 57(13), pp. 3978-3988.
- Lü, Y., Hutchinson, B., Molodov, D.A. and Gottstein, G. (2010) 'Effect of deformation and annealing on the formation and reversion of ε-martensite in an Fe–Mn–C alloy', *Acta Materialia*, 58(8), pp. 3079-3090.
- Lu, Y., Bruhis, M. and McDermid, J.R. (2014) 'Effect of strain path on the work hardening behavior and microstructural evolution of a Fe-Mn-C TWIP steel'. In *Proceedings of HMnS 2014 Conference*, Germany.
- Medvedeva, N.I., Park, M.S., Van Aken, D.C. and Medvedeva, J.E. (2014) 'Firstprinciples study of Mn, Al and C distribution and their effect on stacking fault energies in fcc Fe', *Journal of Alloys and Compounds*, 582, pp. 475-482.
- Meguerian, R. (2007) Hardenability improvements and rate-limiting reactions during hotdip galvanizing of high-Mn dual-phase steels. Master Thesis, McMaster University.
- Mendez, J., Ghoreshy, M., Mackay, W.B.F., Smith, T.J.N. and Smith, R.W. (2004)
 'Weldability of austenitic manganese steel', *Journal of Materials Processing Technology*, 153-154, pp. 596-602.

- Miura, S., Takamura, J. and Narita, N. (1968) 'Orientation dependence of flow stress for twinning in silver crystals'. *Transactions of the Japan Institute of Metals*, 9, pp. 555-562.
- Muèllner, P. and Ferreira, P.J. (1996) 'On the energy of terminated stacking faults', *Philosophical Magazine Letters*, 73(6), pp. 289-298.
- Nakano, J. and Jacques, P.J. (2010) 'Effects of the thermodynamic parameters of the hcp phase on the stacking fault energy calculations in the Fe–Mn and Fe–Mn–C systems', *Calphad*, 34(2), pp. 167-175.
- Nihei, Y., Hiraoka, Y., Kagaya, Y., Kohno, Y., Shibata, K. and Ohtsuka, H. (2004) 'Effect of pre-strain and reheating on isothermal 'αmartensite formation in Fe –25.5Ni–4Cr austenitic steel', *Journal of Nuclear Materials*, 329-333, pp. 648-651.
- Ohkubo, N., Miyakusu, K., Eumatsu, Y. and Kimura, H. (1994) 'Effect of alloying elements on the mechanical properties of the stable austenitic stainless steel', *ISIJ International*, 34(9), pp. 764-772.
- Olson, G.B. and Cohen, M. (1972) 'A mechanism for the strain-induced nucleation of martensitic transformations', *Journal of the Less Common Metals*, 28(1), pp. 107-118.
- Olson, G.B. and Cohen, M. (1976) 'A general mechanism of martensitic nucleation: Part
 I. General concepts and the FC↔HCP transformation', *Metallurgical Transactions A*, 7(12), pp. 1897-1904.
- Petrov, Y.N. (1993) 'On the carbon distribution at structural imperfections in manganese austenite', *Scripta Metallurgica et Materialia*, 29(11), pp. 1471-1476.

- Putaux, J.-L. and Chevalier, J.-P. (1996) 'HREM study of self-accommodated thermal εmartensite in an Fe–Mn–Si–Cr–Ni shape memory alloy', *Acta Materialia*, 44(4), pp. 1701-1716.
- Qin, B. and Bhadeshia, H.K.D.H. (2008) 'Plastic strain due to twinning in austenitic TWIP steels', *Materials Science and Technology*, 24(8), pp. 969-973.
- Remy, L. (1978) 'Kinetics of F.C.C. deformation twinning and its relationship to stressstrain behaviour', *Acta Metallurgica*, 26(3), pp. 443-451.
- Remy, L. and Pineau, A. (1976) 'Twinning and strain-induced F.C.C.→H.C.P. transformation on the mechanical properties of Co–Ni–Cr–Mo alloys', *Materials Science and Engineering*, 26(1), pp. 123-132.
- Remy, L. and Pineau, A. (1977) 'Twinning and strain-induced F.C.C.→H.C.P. transformation in the Fe–Mn–Cr–C system', *Materials Science and Engineering*, 28(1), pp. 99-107.
- Renard, K. and Jacques, P.J. (2012) 'On the relationship between work hardening and twinning rate in TWIP steels', *Materials Science and Engineering: A*, 542, pp. 8-14.
- Renard, K., Ryelandt, S. and Jacques, P.J. (2010) 'Characterisation of the Portevin-Le Châtelier effect affecting an austenitic TWIP steel based on digital image correlation', *Materials Science and Engineering: A*, 527(12), pp. 2969-2977.
- Saeed-Akbari, A., Imlau, J., Prahl, U. and Bleck, W. (2009) 'Derivation and variation in composition-dependent stacking fault energy maps based on subregular solution

model in high-manganese steels', *Metallurgical and Materials Transactions A*, 40(13), pp. 3076-3090.

- Sahu, P., De, M. and Kajiwara, S. (2002) 'Microstructural characterization of Fe–Mn–C martensites athermally transformed at low temperature by Rietveld method', *Materials Science and Engineering: A*, 333(1-2), pp. 10-23.
- Sahu, P., Hamada, A.S., Sahu, T., Puustinen, J., Oittinen, T. and Karjalainen, L.P. (2012)
 'Martensitic transformation during cold rolling deformation of an austenitic Fe-26Mn-0.14C alloy', *Metallurgical and Materials Transactions A*, 43(1), pp. 47-55.
- Sato, A., Soma, K. and Mori, T. (1982) 'Hardening due to pre-existing ε-martensite in an Fe-30Mn-1Si alloy single crystal', *Acta Metallurgica*, 30(10), pp. 1901-1907.
- Sato, K., Ichinose, M., Hirotsu, Y. and Inoue, Y. (1989) 'Effects of deformation induced phase transformation and twinning on the mechanical properties of austenitic Fe-Mn-Al alloys', *ISIJ International*, 29(10), pp. 868-877.
- Schoeck, G. (1999) 'The dissociation energy of extended dislocations in fcc lattices', *Philosophical Magazine A*, 79(5), pp. 1207-1215.
- Schoeck, G. and Seeger, A. (1955) 'Activation energy problems associated with extended dislocations', In *Proceedings of the Conference of Defects in Crystalline Solids*, The Physical Society, London, pp. 340-346.
- Schramm, R.E. and Reed, R.P. (1975) 'Stacking fault energies of seven commercial austenitic stainless steels', *Metallurgical Transactions A*, 6(7), pp. 1345-1351.
- Schumann, H. (1967) 'Die martensitischen umwandlungen in kohlenstoffärmen manganstählen'. *Archive Eisenhüttenwes*, 38, pp. 647-656.

- Schumann, H. (1972) 'Martensitic transformations in austenitic manganese carbon steels, *Neue Huette*, 17(10), pp. 605-609.
- Schumann, H. (1974) 'Influence of stacking fault energy on the crystal lattice change mechanisms in the γ/α phase transformation in high alloy steels', *Kristall und Technik*, 9(10), pp. 1141-1152.
- Shiekhelsouk, M.N., Favier, V., Inal, K. and Cherkaoui, M. (2009) 'Modelling the behaviour of polycrystalline austenitic steel with twinning-induced plasticity effect', *International Journal of Plasticity*, 25(1), pp. 105-133.
- Takaki, S., Furuya, T. and Tokunaga, Y. (1990) 'Effect of Si and Al additions on the low temperature toughness and fracture mode of Fe-27Mn alloys', *ISIJ International*, 30(8), pp. 632-638.
- Takaki, S., Nakatsu, H. and Tokunaga, Y. (1993) 'Effects of austenite grain size on ε martensitic transformation in Fe-15mass%Mn alloy', *Materials Transactions, JIM*, 34(6), pp. 489-495.
- Talonen, J. and Hänninen, H. (2007) 'Formation of shear bands and strain-induced martensite during plastic deformation of metastable austenitic stainless steels', *Acta Materialia*, 55(18), pp. 6108-6118.
- Tsuchida, N., Tomota, Y., Moriya, H., Umezawa, O. and Nagai, K. (2001) 'Application of the Kocks–Mecking model to tensile deformation of an austenitic 25Cr–19Ni steel', *Acta Materialia*, 49(15), pp. 3029-3038.

- Tweedale, G. (1985) 'Sir Robert Abbott Hadfield F.R.S. (1858-1940), and the discovery of manganese steel', *Notes and Records of the Royal Society of London*, 40(1), pp. 63-74.
- Vander Voort, G.F. (1984) Metallography: Principles and Practice. ASM International.
- Venables, J.A. (1961) 'Deformation twinning in face-centred cubic metals', *Philosophical Magazine*, 6(63), pp. 379-396.
- Venables, J.A. (1962) 'The martensite transformation in stainless steel', *Philosophical Magazine*, 7(73), pp. 35-44.
- Volosevich, P.Y., Gridnev, V.N. and Petrov, Y. N. (1976) 'Manganese influence on stacking-fault energy in iron-manganese alloys', *Fizika Metallov i Metallovedenie*, 42(2), pp. 372-376.
- Wang, X., Zurob, H.S., Embury, J.D., Ren, X. and Yakubtsov, I. (2010) 'Microstructural features controlling the deformation and recrystallization behaviour Fe–30% Mn and Fe–30%Mn–0.5%C', *Materials Science and Engineering: A*, 527(16-17), pp. 3785-3791.
- Whelan, M.J. (1959) 'Dislocation interactions in face-centred cubic metals, with particular reference to stainless steel', In *Proceedings of the Royal Society of London*. Series A. Mathematical and Physical Sciences, 249(1256), pp. 114-137.
- Yang, E. (2010) The effect of carbon content on the mechanical properties and microstructural evolution of Fe-22Mn-C TWIP/TRIP steels. Master Thesis, McMaster University, Canada.

- Yang, E., Zurob, H. and McDermid, J. (2010) 'Mechanical behavior and microstructural evolution of Fe-22Mn-C TWIP/TRIP steels as a function of C content', In *Proceedings of MS&T 2010*, United States.
- Yang, J.H. and Wayman, C.M. (1992) 'On secondary variants formed at intersections of ε martensite variants', *Acta Metallurgica et Materialia*, 40(8), pp. 2011-2023.
- Yang, P., Xie, Q., Meng, L., Ding, H. and Tang, Z. (2006) 'Dependence of deformation twinning on grain orientation in a high manganese steel', *Scripta Materialia*, 55(7), pp. 629-631.
- Yoo, J.D., Hwang, S.W. and Park, K.-T. (2009) 'Origin of extended tensile ductility of a Fe-28Mn-10Al-1C steel', *Metallurgical and Materials Transactions A*, 40(7), pp. 1520-1523.



Appendix A: Repeatability of Tensile Results for the Experimental Alloys

(b) Fe-22Mn-0.4C alloy

Figure A-1: Room temperature true stress-true strain curves for the homogenous Fe-22Mn-0.2C and Fe-22Mn-0.4C alloys.



Figure A-2: Room temperature true stress-true strain curves for the homogenous Fe-22Mn-0.6C and carbon graded G1 alloys.



Figure A-3: Room temperature true stress-true strain curves for the carbon graded G2 and G3 alloys.



(b) Fe-30Mn-0.4C alloy

Figure A-4: Room temperature true stress-true strain curves for the homogenous Fe-30Mn-0.2C and Fe-30Mn-0.4C alloys.



(b) carbon graded Fe-30Mn-C alloy

Figure A-5: Room temperature true stress-true strain curves for the homogenous Fe-30Mn-0.6C and carbon graded Fe-30Mn-C alloys.

Appendix B: Microstructural Evolution Results for the Carbon Graded G1

Alloy



Figure B-1: EBSD phase maps from the near surface cross-section (0-250 microns from the surface) of G1 alloy as a function of true strain.



Figure B-2: EBSD phase maps from the mid-thickness cross-section (250-500 microns from the surface) of G1 alloy as a function of true strain.



Ph.D. Thesis – M. Ghasri Khouzani

Figure B-3: EBSD phase maps from the core cross-section (500-750 microns from the surface) of G1 alloy as a function of true strain.

Appendix C: Fractography Results for the Carbon Graded G1 Alloy



(a) near surface (0-250 μ m from the surface)

(b) mid-thickness (250-500 μ m from the

surface)



(c) core (500-750 μ m from the surface)





(a) near surface cross-section (0-250 μ m from

the surface)



(b) mid-thickness cross-section (250-500 µm

from the surface)



(c) core cross-section (500-750 μ m from the surface)

Figure C-2: SEM micrographs from cross-section near fracture surface, sectioned along

the tensile direction, of the carbon graded G1 alloy.