The Influence of Silicon and Aluminum Alloying on Austenitic Steel Lattice Parameter and Stacking Fault Energy

G.R. Lehnhoff, K.O. Findley, B.C. De Cooman

Abstract

The influence of Si and Al on austenitic steel lattice parameter and stacking fault energy (SFE) were studied. X-ray diffraction indicated that 2.5 wt pct Al alloying increased the austenite lattice parameter by 1.41×10^{-3} nm, while 2.5 wt pct Si alloying had little effect. Partial dislocation separation measurements from weak beam dark field images indicated that 2.5 wt pct Al alloying increased the SFE by 12-17 mJ/m² and 2.5 wt pct Si alloying decreased the SFE by 6-7 mJ/m².

Key words: Metastable austenitic steel; Stacking fault energy; Transmission electron microscopy (TEM); Lattice parameter

Advanced high strength steels (AHSS) containing metastable austenite are candidate materials for automobile weight reduction. Example materials include transformation induced plasticity (TRIP) steels [1,2], low alloy quench and partitioning (Q&P) steels [3-5], and more highly Mn-alloyed third generation AHSS [6-8]. Recent investigations regarding TRIP steels have evaluated the substitution of Al for Si alloying. Both Si and Al inhibit carbide formation during austempering and promote austenite retention, but Al alloying accelerates the bainitic transformation during austempering [9,10] and results in better coatability [11,12]. Al alloying in TRIP steels has been shown to increase resistance to strain induced α' martensitic transformation relative to Si-alloyed grades [13,14]. Explanations for the observed increase in austenite stability with Al alloying include increases in carbon partitioning to austenite during austempering [14,15] and increases in austenite stacking fault energy (SFE) [13]. This paper will focus on the effects of Al and Si alloying on the measured austenite carbon content and SFE.

Carbon is a potent austenite stabilizer, and its concentration in retained austenite is commonly ascertained by X-ray diffraction (XRD) measurements of the lattice parameter. Carbon is known to expand the austenite lattice, and linear correlations between austenite lattice parameter (a_o) and carbon content are proposed in the literature [16-19]. However, Al is also reported to expand the austenite lattice [16]. Often, the influence of Al on the austenite lattice parameter is excluded when calculating the austenite C-content. This exclusion may lead to overestimations of the austenite C-content in Al-alloyed AHSS steels, and thus may be misleading in the effect of Al on carbon partitioning to austenite. In contrast, Si is reported to have a negligible influence on the austenite lattice parameter [16].

The SFE also affects austenite stability through its effect on shear band formation. Strain induced α' martensite nucleation can occur on the intersection of planar shear bands [20]. A higher austenite SFE is expected to promote dislocation cross slip, which inhibits shear band formation and martensite nucleation. Thus, Al alloying may stabilize austenite by increasing the austenite SFE [21-24]. The effect of Si on the austenite SFE is less clear. Experimental TEM [25] and XRD [26-28] studies indicate that Si decreases the SFE of austenite, while thermochemical modeling indicates that Si slightly decreases the SFE [29] or that the influence of Si on the SFE is non-monotonic [21].

In this study, the influences of Si and Al on the austenite lattice parameter and SFE are investigated using the three austenitic steel compositions shown in Table 1, which were derived from a microstructure survey of the Fe-Ni-Cr-Al-Si system [30]. The compositions are all approximately the same except for variations in Al and Si alloying, which allows the effects of these elements on lattice parameter and SFE to be directly assessed. The three steels were vacuum cast, homogenized for 20 hours at 1200 °C, hot rolled, and then annealed for one hour at the temperatures indicated in Table 1. The annealing temperatures were chosen to achieve coarse grain sizes of approximately 140 μm .

Fully austenitic steels were employed because no assumptions regarding element partitioning in a multiphase microstructure have to be made in the XRD analysis. Furthermore, transmission electron microscopy (TEM) analysis of dislocations for SFE determination is performed more easily with a coarse grain, single phase microstructure. However, the assumptions and possible limitations of extending the analysis to lower alloy multiphase AHSS must be acknowledged. Austenite is retained to room temperature in the experimental alloys by Ni and Cr alloying, not by carbon partitioning, as in AHSS. Therefore, to extend the results of this study to low alloy AHSS, the effects of Si and Al on austenite characteristics are assumed independent of Ni and Cr alloying.

Table 1. Experimental Steel Compositions and Solution Annealing Temperatures.

Seven transverse coupons for XRD examination were taken from various locations in the hot rolled and annealed plates for each material. The coupons were ground to a 600 grit surface finish and then chemically polished for 20 minutes in a solution of 10 parts water, 10 parts hydrogen peroxide, and 1 part hydrofluoric acid. A Phillips X'Pert® diffractometer with $\text{CuK}\alpha$ ($\lambda = 0.154060 \text{ nm}$) radiation was used for XRD. HighScore Plus® 3.0.5 software was used to subtract signal backgrounds, strip $\text{CuK}\alpha_2$ peaks from the scans, perform profile fitting, and determine the peak positions (2θ) and corresponding interplanar spacings. The lattice parameters (a_0) determined from the first four austenite peaks ((111), (200), (220), and (311)) were plotted against $\cos^2\theta/\sin\theta$, and a_0 for each specimen was determined from a linear regression extrapolation to $\theta = 90^\circ$ [31]. The a_0 versus $\cos^2\theta/\sin\theta$ regression analysis was weighted

to favor higher angle peaks by using quadruplicate data points for (311) peaks, triplicate data points for (220) peaks, duplicate data points for (200) peaks, and single data points for (111) peaks [32]. The lattice parameter for each material was then obtained by discarding the lowest and highest values of a_0 , and averaging the results of the remaining five specimens.

For the SFE determination, 12.7 mm diameter cylinders of each material were machined with the cylinder axes parallel to the rolling direction. The cylinders were compressed to 5 pct true strain to introduce dislocations into the specimens. Thin slices were then sectioned from the cylinders at 45° from the compression axis and ground to a final thickness of approximately 140 μm with a 600 grit surface finish on both sides. The slices were then punched into 3 mm diameter disks and twin jet electropolished until perforation in a solution of 95 pct acetic acid and 5 pct perchloric acid. Electropolishing was done at room temperature with a voltage of 25-50 V and current of 30-35 mA.

TEM investigation of the electropolished disks was performed in a Phillips CM12® TEM operating at 120 kV. Diffraction rotation was corrected using elongated crystals of molybdenum trioxide [33,34]. For each material, grains with $\{111\}$ planes nearly aligned with the plane of the foil were used for weak beam dark field (WBDF) imaging of dissociated dislocations [33,34]. The WBDF imaging was performed using $\mathbf{g}3\mathbf{g}$ conditions with (220) type reflections ($s_g \approx 0.22 \text{ nm}^{-1}$). Grains near a $\{111\}$ orientation were selected because they had favorable slip conditions during compression and would therefore contain dislocations. Furthermore, dissociated dislocations imaged in this configuration are minimally inclined to the electron beam. Thus, the measured partial dislocation separations do not need to be corrected for 2D image projection. The evaluated dislocations were confirmed to exist in the $\{111\}$ plane approximately normal to the electron beam through $\mathbf{g} \cdot \mathbf{b}_p$ analysis of the partial dislocations [33,34].

For each material, multiple data pairs of partial dislocation separation, d , and perfect dislocation character angle, α , were recorded along several dislocations using ImageJ software. Figure 1 shows an example WBDF image of a dissociated dislocation in the Base alloy, along with representations of d and α . As shown in Figure 1, α is the angle between the line vector and the Burgers vector, $\mathbf{b}$, of the undissociated dislocation. Because both partial dislocations are in contrast in the condition shown and because $\mathbf{g} \cdot \mathbf{b}_p$ analysis was performed, it is known that the $\mathbf{g}$ vector used to form the two beam condition is parallel to the undissociated dislocation Burgers vector, $\mathbf{b}$. The paired (d , α) data were then input into Equation 1 [35] to calculate SFE values for each measured data pair:

$$SFE = \frac{G|a_0|^2(2-\nu)}{48\pi(1-\nu)} \cdot \frac{1}{d} \cdot \left(1 - \frac{2\nu \cos 2\alpha}{2-\nu}\right) \quad (1)$$

These values were then averaged to determine the SFE for each material. The experimental lattice parameters (a_0) determined from the XRD analysis were used in Equation 1. The isotropic shear modulus (G) and Poisson ratio (ν) were assumed to be the same for each material and were 74 GPa and 0.3 for

shear modulus and Poisson ratio, respectively [36]. In order to estimate the possible effect of elastic anisotropy on $\{111\}$ dislocations, the effective G (66 GPa) and effective ν (0.43) values determined for a 12 Ni – 12 Cr steel [37,38] were also applied to the experimental WBDF data. Use of the effective elastic constants increased the calculated SFE value for the 2.5 Al alloy by approximately 10 pct, but had little effect on the other two alloys. Since elastic anisotropy was not evaluated in this study and since other literature reports an effective ν closer to 0.3 instead of 0.43 [29], the bulk elastic properties were used in the SFE calculations.

Figure 1. Weak beam dark field TEM micrograph of a dissociated dislocation in the Base alloy. Annotations are explained in the text.

Figure 2 and Table 2 show the calculated austenite lattice parameter as a function of relative Si and Al alloying. Relative Si alloying refers to the difference in Si content between the 2.5 Si and the Base alloys and relative Al alloying refers to the difference in Al content between the 2.5 Al and Base alloys. Because all of the alloys have approximately equal contents of all elements other than Si and Al (Table 1), only differences in Si and Al content have been considered. Figure 2 indicates that 2.5 wt pct Al alloying increases the austenite lattice parameter by 1.41×10^{-3} nm relative to the Base alloy, while 2.5 wt pct Si alloying has a minimal effect. Linear empirical relations between austenite composition and lattice parameter for austenitic steels are reported in the literature [16] and take the form of $a_0 = \sum_i A_i^{LP} x_i$ where x_i is the wt pct of element i in austenite and A_i^{LP} is the linear lattice parameter coefficient for element i with units of 10^{-3} nm/wt pct. The calculated A^{LP} for Si and Al are -0.018 and 0.582×10^{-3} nm/wt pct, respectively. These values agree well with those reported by Dyson and Holmes (Table 2).

Figure 2. The dependence of austenite lattice parameter on Si and Al alloying.

Table 2. Empirical Si and Al Linear Elemental Coefficients for Austenite Lattice Parameter and Stacking Fault Energy.

Since Al alloying increases the austenite lattice parameter, it is important to account for Al in calculations of the austenite C-content based on the lattice parameter. If only carbon is considered to expand the lattice, the calculated C-content may be an overestimate. Values for A_C^{LP} reported in the literature range from as low as 3.30×10^{-3} nm/wt pct for austenitic stainless steels to as high as 4.67×10^{-3} nm/wt pct for low alloy steels [16-19]. If Al alloying is neglected, the overestimate of austenite C-content, δ_C , can be expressed as $\delta_C = x_{Al} (A_{Al}^{LP} / A_C^{LP})$. Al-alloying in TRIP steels is often on the order of 1 wt pct.

Using the value of A_{Al}^{LP} found in this study, the range of A_C^{LP} values reported in the literature, and assuming x_{Al} is 1 wt pct, the value of δ_C ranges from 0.125 to 0.176 wt pct.

The magnitude of δ_C is non-negligible. An increase in retained austenite carbon content in multiphase TRIP steels of 0.09 to 0.21 wt pct has been shown to increase its thermodynamic stability [39]. This carbon content range is on the same order of magnitude of the δ_C estimations above. Thus, the influence of Al on the measured retained austenite lattice parameter should not be ignored. It should also be noted that the actual value of δ_C for AHSS depends on the nominal steel composition and thermal processing schedule. For example, in intercritically annealed TRIP steel, Al may partition to the intercritical ferrite, resulting in retained austenite Al-content, x_{Al} , that is lower than the nominal steel Al-content. Conversely, if the steel is instead fully austenitized before lower temperature carbon partitioning, the Al-content of the austenite retained to room temperature should equal the nominal steel composition because limited substitutional element partitioning is expected during lower temperature carbon partitioning [40].

Composition also influences austenite stability through its effect on the thermodynamics of the austenite-to-epsilon martensite transformation and the SFE. Lower austenite SFE values promote the formation of planar shear bands, including epsilon martensite, whose intersections may serve as α' martensite nuclei [20]. Increasing the austenite SFE is expected to suppress shear band formation and strain induced martensite formation. Therefore, it is important to characterize the effects of Al and Si alloying on the SFE.

Figure 3(a) shows the measurements of partial dislocation spacing as a function of dislocation angle for the Base, 2.5 Si, and 2.5 Al alloys. In Figure 3(a), the solid lines represent the average calculated SFE for each material and the dashed lines indicate the upper and lower bounds from the calculated SFE standard deviations. The Base alloy SFE is 26.3 mJ/m², which is consistent with the value of 30 mJ/m² found for a similar 15 Ni – 11 Cr alloy through TEM analysis of extended dislocation nodes [25]. Figure 3(b) shows the dependence of SFE on relative Si and Al alloying. Figure 3(b) indicates that 2.5 wt pct Al alloying increases the SFE by 12.0 mJ/m² while 2.5 wt pct Si alloying decreases the SFE by 6.4 mJ/m² from the Base alloy value. A correction is available to account for the effect of the partial dislocation strain fields on the observed partial dislocation spacings, and thus the measured SFE values [34]. The correction suggested by Williams and Carter was not applied because all of the TEM data was not available to assign all of the necessary parameters. An upper limit estimation suggests that the biggest change is in the 2.5 Al alloy, where the correction would increase the SFE of the 2.5 Al alloy to 44.0 ± 9.8 mJ/m². The SFEs of the other alloys would change by approximately 1 mJ/m² or less. Since the corrected values are estimated, the uncorrected data will be used for the remainder of the paper unless otherwise noted.

Figure 3. (a) Dependence of partial dislocation separation on perfect dislocation character angle for the Base, 2.5 Si, and 2.5 Al alloys. Symbols indicate measured data while the solid lines indicate the calculated trends based on the calculated average SFE for each material. The dashed lines indicate the upper and lower bounds determined from the calculated SFE standard deviation for each material. (b) Dependence of stacking fault energy on Si and Al alloying.

Al has also been reported in literature to increase the SFE [21-24], which is consistent with the current observations. However, there are conflicting reports of the effect of Si on austenite SFE; some sources indicate Si decreases the SFE [25-29], which is supported by the current study, while others suggest the dependence of the SFE on Si alloying is non-monotonic [21]. Linear empirical relationships between austenite composition and SFE have been proposed [25,26] and take the form of $SFE = \sum_i A_i^{SFE} x_i$, where x_i is the wt pct of element i in austenite and A_i^{SFE} is the linear SFE coefficient for element i with units of $\text{mJ/m}^2/\text{wt pct}$. Table 2 contains the values of A_{Si}^{SFE} and A_{Al}^{SFE} found in this study.

Values of A_{Si}^{SFE} reported in the high-Mn steel literature vary from -1 to -3.5 $\text{mJ/m}^2/\text{wt pct}$ [27-29], which is in good agreement with the value of -2.6 $\text{mJ/m}^2/\text{wt pct}$ found in this study (Table 2). Thermochemical modeling of the SFE in high-Mn alloys predicts an A_{Al}^{SFE} value of approximately 5.2 $\text{mJ/m}^2/\text{wt pct}$ [21], which is in very good agreement with the value of 4.9 $\text{mJ/m}^2/\text{wt pct}$ found in this study (Table 2). Jeong *et al.* found an A_{Al}^{SFE} value of approximately 6.4 $\text{mJ/m}^2/\text{wt pct}$ in high-Mn alloys using XRD analysis [28]. Considering the influence of the partial dislocation strain fields on their apparent position (and the calculated SFE) for the Base and 2.5 Al alloys yields an estimated A_{Al}^{SFE} value of 6.9 $\text{mJ/m}^2/\text{wt pct}$ in this study. In contrast, Kim *et al.* showed by TEM WBDF imaging of dissociated dislocations that Al increases the SFE in high-Mn steels by 11.3 $\text{mJ/m}^2/\text{wt pct}$ [23], which is higher than the values found in this study.

In conclusion, it was found that 2.5 wt pct Al alloying increases the austenite lattice parameter by 1.41×10^{-3} nm and Si has little effect. Furthermore, ignoring the effects of Al alloying on austenite lattice parameter may result in an overestimation of the C content on the order of 0.15 wt pct in Al-alloyed AHSS. Finally, 2.5 wt pct Al alloying increases SFE by 12-17 mJ/m^2 and 2.5 wt pct Si alloying decreases the SFE by 6-7 mJ/m^2 in the alloy system investigated. These observations are generally supported by other reports of alloying effects on SFE.

The support of the Advanced Steel Processing and Products Research Center (ASPPRC), the National Science Foundation CAREER Award No. 0955236, and the US Department of Energy Office of Science Graduate Fellowship Award are gratefully acknowledged.

- [1] O. Matsumura, Y. Sakuma, H. Takechi, *Scr. Metall.* 21 (1987) 1301.
- [2] B.C. De Cooman, *Curr. Opin. Solid State & Mater. Sci.* 8 (2004) 285.
- [3] J. Speer, D.K. Matlock, B.C. De Cooman, J.G. Schroth, *Acta Mater.* 51 (2003) 2611.
- [4] J.G. Speer, D.V. Edmonds, F.C. Rizzo, D.K. Matlock, *Curr. Opin. Solid State & Mater. Sci.* 8 (2004) 219.
- [5] D.V. Edmonds, K. He, F.C. Rizzo, B.C. De Cooman, D.K. Matlock, J.G. Speer, *Mater. Sci. & Eng. A* 438-40 (2006) 25.
- [6] M.J. Merwin, *Mater. Sci. Forum* 539-43 (2007) 4327.
- [7] E. De Moor, J.G. Speer, D.K. Matlock, J.-H. Kwak, S.-B. Lee, *ISIJ Int.* 51 (2011) 137.
- [8] P.J. Gibbs, E. De Moor, M.J. Merwin, B. Clausen, J.G. Speer, D.K. Matlock, *Metall. & Mater. Trans. A* 42 (2011) 3691.
- [9] J. Mahieu, J. Maki, B.C. De Cooman, S. Claessens, *Metall. & Mater. Trans. A* 33 (2002) 2573.
- [10] A. Mertens, P.J. Jacques, L. Zhao, S.O. Kruijver, J. Sietsma, F. Delannay, *J. Phys. IV France* 112 (2003) 305.
- [11] J. Mahieu, S. Claessens, B.C. De Cooman, *Metall. & Mater. Trans. A* 32 (2001) 2905.
- [12] E.M. Bellhouse, J.R. McDermid, *Mater. Sci. & Eng. A* 491 (2008) 39.
- [13] L. Samek, E. De Moor, J. Penning, B.C. De Cooman, *Metall. & Mater. Trans. A* 37 (2006) 109.
- [14] A.K. De, R.S. Kircher, J.G. Speer, D.K. Matlock, in: *Proc. Int. Conf. on Adv. High-Strength Sheet Steels for Automot. Appl.*, ed. J.G. Speer, AIST, Warrendale, PA, 2004, pp. 337.
- [15] S. Taint, A. Pichler, K. Hauzenberger, P. Stiaszny, E. Werner, *Steel Res. Int.* 73 (2002) 259.
- [16] D.J. Dyson, B. Holmes, *J. Iron Steel Inst.* 208 (1970) 469.
- [17] C. S. Roberts, *Trans. TMS-AIME* 197 (1953) 203.
- [18] N.H. van Dijk, A.M. Butt, L. Zhao, J. Sietsma, S.E. Offerman, J.P. Wright, S. van der Zwaag, *Acta Mater.* 53 (2005) 5439.
- [19] Z. Nishiyama, *Martensite Transformation*, Maruzen, Tokyo, (1979).
- [20] G.B. Olson, M. Cohen, *Metall. Trans. A* 6 (1975) 791.
- [21] A. Dumay, J.-P. Chateau, S. Allain, S. Migot, O. Bouaziz, *Mater. Sci. & Eng. A* 483-4 (2008) 184.
- [22] W.S. Yang, C.M. Wan, *J. Mater. Sci.* 25 (1990) 1821.
- [23] J. Kim, S.-J. Lee, B.C. De Cooman, *Scripta Mater.* 65 (2011) 363.
- [24] J. Kim, B.C. De Cooman, *Metall. & Mater. Trans. A* 42 (2011) 932.
- [25] C.G. Rhodes, A.W. Thompson, *Metall. Trans. A* 8 (1977) 1901.
- [26] R.E. Schramm, R.P. Reed, *Metall. Trans. A* 6 (1975) 1345.
- [27] X. Tian, Y. Zhang, *Mater. Sci. & Eng. A* 516 (2009) 73.
- [28] K. Jeong, J.-E. Jin, Y.-S. Jung, S. Kang, Y.-K. Lee, *Acta Mater.* 61 (2013) 3399.
- [29] D.T. Pierce, J.A. Jimenez, J. Bentley, D. Raabe, C. Oskay, J.E. Wittig, *Acta Mater.* 68 (2014) 238.
- [30] M.L. Glenn, *J. Mater. Eng.* 10 (1988) 181.
- [31] B.D. Cullity, S.R. Stock, *Elements of X-Ray Diffraction*, third ed., Prentice Hall, New Jersey, 2001.
- [32] C. Garcia-Mateo, F.G. Caballero, M.K. Miller, J.A. Jimenez, *J. Mater. Sci.* 47 (2012) 1004.
- [33] M.H. Loretto, R.E. Smallman, *Defect Analysis in Electron Microscopy*, Chapman & Hall, London, 1975.
- [34] D.B. Williams, C.B. Carter, *Transmission Electron Microscopy*, second ed., Springer, New York, 2009.
- [35] W.T. Read, *Dislocations in Crystals*, McGraw-Hill, New York, 1953.
- [36] B.C. De Cooman, J.G. Speer, *Fundamentals of Steel Product Physical Metallurgy*, AIST, 2011.
- [37] K. Salmutter, F. Stangler, *Z. Metallk.* 51 (1960) 544.
- [38] R.M. Latanision, A.W. Ruff Jr., *Metall. Trans.* 2 (1971) 505.
- [39] E. Jimenez-Melero, N.H. van Dijk, L. Zhao, J. Sietsma, S.E. Offerman, J.P. Wright, S. van der Zwaag, *Acta Mater.* 57 (2009) 533.
- [40] I. Stark, G.D.W. Smith, H.K.D.H. Bhadeshia, *Metall. Trans. A* 21 (1990) 837.

Figure 1

Figure 1. Weak beam dark field TEM micrograph of a dissociated dislocation in the Base alloy. Annotations are explained in the text.

Figure 2

Figure 2. The dependence of austenite lattice parameter on Si and Al alloying.

Figure 3

Figure 3. (a) Dependence of partial dislocation separation on perfect dislocation character angle for the Base, 2.5 Si, and 2.5 Al alloys. Symbols indicate measured data while the solid lines indicate the calculated trends based on the calculated average SFE for each material. The dashed lines indicate the upper and lower bounds determined from the calculated SFE standard deviation for each material. (b) Dependence of stacking fault energy on Si and Al alloying.

Table 1. Experimental Steel Compositions and Solution Annealing Temperatures.

wt pct	C	N	Ni	Cr	Mn	Si	Al	Annealing Temp. (°C)
Base	0.028	0.0034	15.05	11.06	1.09	0.002	0.05	1040
2.5 Si	0.028	0.0036	14.99	10.98	1.10	2.50	0.06	1075
2.5 Al	0.030	0.0034	15.04	11.02	1.08	0.034	2.47	1060

Table 2. Empirical Si and Al Linear Elemental Coefficients for Austenite Lattice Parameter and Stacking Fault Energy.

Element	A^{LP} (10^{-3} nm/wt pct) [10^{-3} nm/at pct]	A^{LP} (10^{-3} nm/wt pct) [10^{-3} nm/at pct] Dyson and Holmes [16]	A^{SFE} (mJ/m ² /wt pct) [mJ/m ² /at pct]
Si	-0.018 [-0.009]	≈ 0 [≈ 0]	-2.6 [-1.3]
Al	0.582 [0.289]	0.56 [0.28]	4.9 [2.5]