FUNDAMENTAL STUDY OF THE AUSTENITE FORMATION AND DECOMPOSITION IN HIGH STRENGTH LOW-Si, Al ADDED Nb-Mo TRIP STEELS

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ABSTRACT

TRIP (Transformation Induced Plasticity) steels are under development for automotive applications that require high strength and high formability. In this study, the formation and decomposition behavior of austenite from the intercritical (α+γ) region in a series of 0.15C-0.3Si-0.15Mo-0.03Nb steels with and without Al additions were evaluated. In addition, the systematic study of the recrystallization of ferrite during intercritical annealing was also investigated. The effect of the initial hot band microstructure on the formation of intercritically annealed austenite and the decomposition products of austenite during different thermal cooling paths from the intercritical region were defined and quantified. The resulting microstructures exhibited tensile strengths in excess of 790 MPa with elongations equal to or better than 25%. The results of this investigation will be presented and discussed in this paper.

KEYWORDS:

TRIP Steel, epitaxial ferrite, retained austenite, bainite, EBSD, Image Quality, CG Processing

INTRODUCTION

High strength sheet steel grades have attracted the interest of the automotive industry because of the necessity to reduce car weight and to improve the safety of cars. The transformation induced plasticity mechanism of TRIP steels represents a very promising approach to increase the ductility of high strength steels grades. In TRIP steels metastable austenite is transformed into martensite during deformation processes such as forming and stretching, thus yielding an outstanding uniform elongation and formability at a very high strength level.

Unfortunately, there are various factors that hinder the application of high strength steels sheets for automotive parts, such as high production costs, poor formability, weldability problems, and the hot dip Zn coating. Some of these problems are associated with the high C and Si content of conventional TRIP steels. Therefore, the reduction of the C and Si content and a partial or complete substitution of Si by other elements are of a major importance in the development of advanced low-alloyed TRIP steels.

In this work, the effect of Al content, coiling temperature and intercritical annealing temperatures were investigated to study: 1) the formation of austenite and the recrystallization of ferrite and 2) the decomposition products of austenite from the intercritical annealing temperature and during the isothermal holding at 450°C.

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In addition, it was also of great interest to evaluate of the amount and composition of retained austenite and its stability through its decomposition from the intercritical annealing temperature as a function of a) cooling rate from the T_{IA} to the isothermal holding temperature, b) holding time and c) cooling rate from the isothermal holding temperature to room temperature. Finally, this paper correlates the processing routes with the observed microstructures and mechanical properties, in order to provide guidelines to develop a commercial 800 MPa low silicon, Al added TRIP steel that can be produced by CGL processing.

EXPERIMENTAL PROCEDURE

Three TRIP steel alloys were investigated, and their Al content varied from 0.05Al to 1.0Al wt percent. Furthermore, constant microalloying additions of Nb and Mo were added to refine the microstructure and to increase strength. The laboratory vacuum melted slab chemistry analyses of the three steels used in this research work are shown in Table I.

Table I. Chemical Composition of TRIP Steels Used in this Study, wt%

Element / Designation	C	Mn	Si	Al	Nb+Mo
0.05Al	0.15	1.52	0.31	0.05	X
0.5Al	0.15	1.52	0.31	0.5	X
1.0Al	0.15	1.52	0.31	1.0	X

Different thermomechanical schedules were performed in order to obtain different hot band microstructures. Based on previous work [1,2], it was determined that the variation of coiling temperature would be sufficient to obtain a large variation in the starting microstructures. Table II shows the design of the temperatures used in the TMP.

Table II. Experimental Design of Thermomechanical Processing

TMP stage	**Temperature**	**Experimental Details**
Reheating Temperature	1250°C	1 hour
Roughing Temperature	1050°C	Two pass @ ε=50% each
Finishing Temperature	950°C	1 pass at ε=50%,
Coiling Temperature	700°C, 550°C	Furnace cooled (30°C/hr)

The hot bands were then cold rolled 60% in several passes. The J-Mat Software was used to predict the intercritical annealing temperatures that would give 20-80% initial austenite for the different alloys. . The samples were heated at 3°C/sec and intercritically annealed for 60 seconds at various temperatures followed by quenching in ice brine solution. The austenite and ferrite volume fractions were then measured to compare the predicted vs observed results.

In order to evaluate the effect of varying intercritical annealing temperatures and cooling rates to the IBT, it was necessary to develop microstructural maps a) during cooling from the T_{IA} to IBT (CCT diagrams) and b) during the isothermal holding temperature IHT (TTT diagrams). A Gleeble 3500 unit fitted with a laser extensometer was used to study the transformation behavior of austenite during continuous cooling from two intercritical annealing temperatures that represented an initial austenite percent of 35% (i.e., low T_{IA}) and 55% (i.e., high T_{IA}). Similarly, in order to study and quantify the austenite decomposition products during cooling from the intercritical annealing temperature to the isothermal holding temperature (IHT), interrupted quench experiments were carried out in a MTS-458 unit with a mounted radiation furnace. During these experiments, samples were also heated at a constant rate of 3°C/sec to the intercritical annealing temperature (equal to 35% and 55% austenite percent), held for 60 seconds and cooled at 5 and 15°C/sec to the isothermal holding temperature of 450°C, followed by a 2-3 second holding and immediately quenched into iced brine solution.

Finally, the holding times of 2s, 30s, 60s, 90, and 120s at the isothermal transformation temperature of 450°C were investigated. Furthermore, the cooling rate to room temperature after each holding time was varied in order to study the effect of post-holding cooling on the retained austenite stability.

<u>Experimental Techniques/Microstructural Analysis</u>

Specimens were sectioned through the thickness, parallel to the rolling direction. Optical microscopy was used for the initial examination and identification of the different types of microstructures. LePera's [3,4] and 10% $Na_2S_2O_5$ [5,6] techniques, were found to give satisfactory results for the characterization of the microconstituents. A Philips XL30 field emission gun SEM and Electron Back Scattering Diffraction (EBSD)-IQ (image quality) filtered grain boundary analysis [7] was used for a more comprehensive and detailed identification of microconstituents. TEM examination of the specimens was conducted using a JEM-200CX electron microscope operated at 200 kV. The microstructural features that were studied included examination and identification of the different phases and their distribution and morphology, and also for the presence of fine precipitates. From XRD-determined lattice parameter of austenite, a_γ, the carbon content of retained austenite was estimated as:

$$C_\gamma = \frac{a_\gamma - 3.5467}{0.0467} \tag{1}$$

where a_γ is in angstroms and C_γ is in weight percent [8]. Finally, Magnetometry performed in a vibrating specimen magnetometer (VSM) was the preferred method for obtaining retained austenite volume fraction.

For the evaluation of the tensile properties, mechanical testing was performed on a MTS 880 unit using ATSM sub-size tensile specimens obtained from CGL simulations on a Gleeble 3500 unit at the U.S. Steel Technical Center.

RESULTS

<u>a) Intercritical Annealing</u>

Three major events take place during the intercritical annealing: dissolution of Fe_3C, recrystallization of the cold rolled ferrite and the formation of austenite. This study shows that the variation of Al content has a large effect on the microstructural characteristics of the austenite and ferrite at any given temperature during intercritical annealing.

It was observed that in the 0.05Al alloy, a prior coiling temperature of 550 °C results in a ~15% (absolute value) larger volume fraction of martensite (austenite) when intercritically annealed at

780°C compared to the sample prior coiled at 700°C. However, in the 0.5Al and 1.0Al, no effect of the prior coiling conditions on the volume fraction of austenite was observed. This is believed to be because in those conditions, the variation in coiling temperature did not result in different starting types of ferrites. On the other hand, the variation in the coiling temperature in the 0.05Al did result in a significant change in the starting type of ferrite in the hot band, from polygonal ferrite when coiled at 700°C, to acicular ferrite when coiled at 550°C. The critical microstructural parameter here is thought to be the change in cementite distribution with coiling temperature.

A similar trend was observed with respect to the recrystallization behavior as a function of prior coiling temperature. However, there was a large influence on both the recrystallization of ferrite and volume fraction of austenite with variation in Al additions in the TRIP alloy. Figure 1 shows a J-Mat Pro simulation of the volume fraction of austenite for the three Al-added TRIP alloys. Measurements of the volume fraction of austenite had a very close agreement with those values suggested by J-Mat Pro. The recrystallization of ferrite was found to be much faster in the 1.0Al alloy than the 0.5Al and 0.05Al alloy. Measurements of the aspect ratio of ferrite grains indicate that recrystallization takes place simultaneously with the formation of austenite and slows down after reaching values close to 75% austenite formation in the 0.05Al and 0.5Al, where as in the 1.0Al recrystallization is complete at a very early stage during intercritical annealing, just before reaching 25% austenite formation. However, it must be pointed out that the IQ analysis indicated that recrystallization takes place during heating and is independent of intercritical annealing temperature or holding time.

Table III shows the amount of austenite in addition to the amounts of non-recrystallized and recrystallized ferrite at various temperatures for the 0.05Al and 1.0Al alloys obtained by IQ analysis. Similarly, the normalized recrystallized ferrite, which is the amount of recrystallized ferrite divided by the total amount of ferrite at any given temperature, is shown in this table. Figure 2 shows the micrographs of the TRIP alloys after intercritical annealing for an initial austenite volume fraction of 35%.

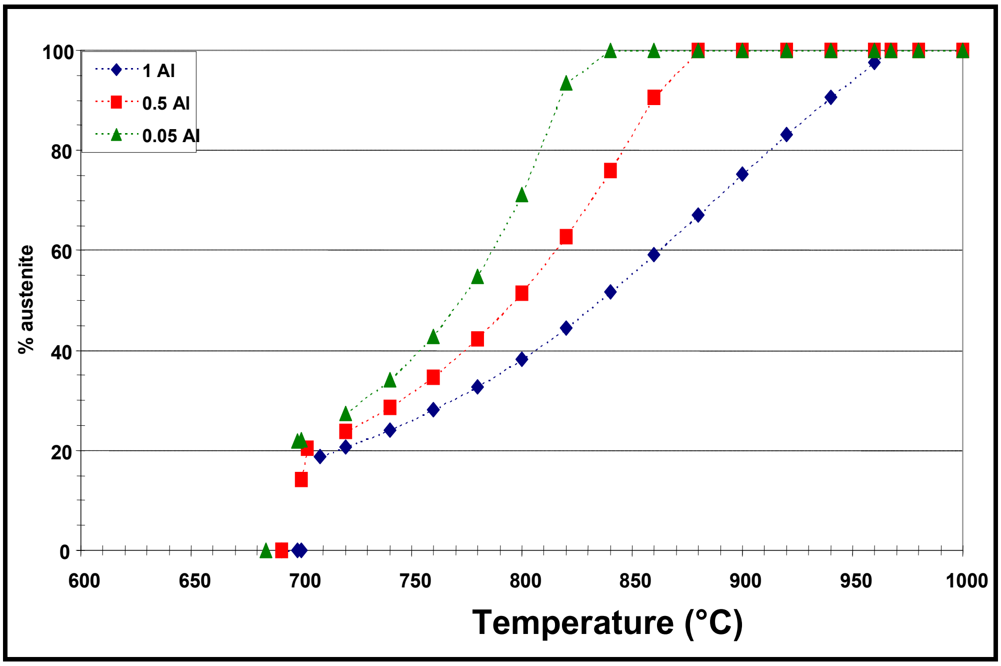

Figure 1: J-Mat Pro simulation of the effect of Al wt% on the austenite volume fraction

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(a) (b) (c)

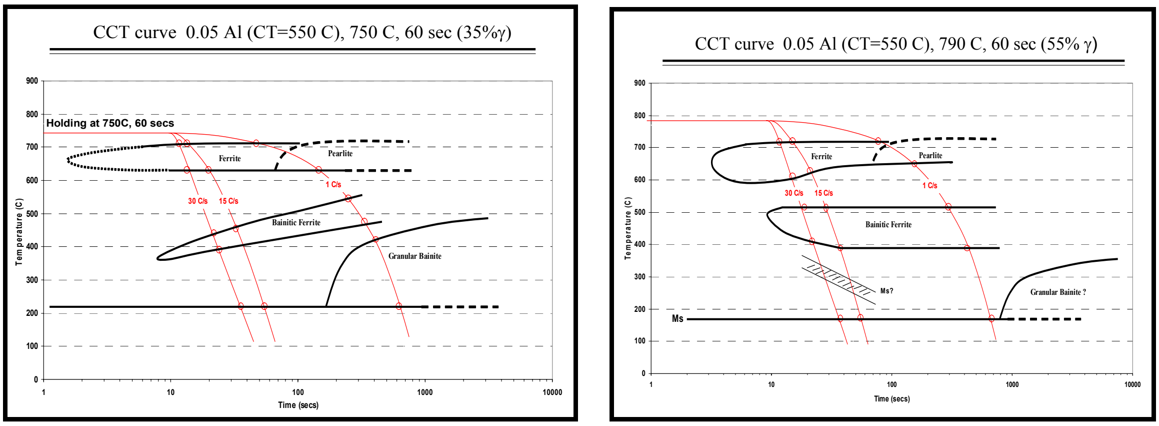
Figure 2: a) 0.05Al alloy, T_{IA}=750°C (35%γ), b) 0.5Al alloy, T_{IA}=760°C (35%γ), c) 1.0Al alloy, T_{IA}=770°C (35%γ). Prior coiling temperature=550°C

Table III. Quantitative Microstructural IQ Analysis of Specimens Intercritically Annealed to Obtain an Initial γ=35% and γ=55%, Followed by quench, in 0.05Al-550 and 1.0Al-550 Alloys

Alloy + T_{IA} / **Microstructures**	**0.05Al-550**		**1.0Al-550**	
	750 C, 60s, Quench (35%γ, Point Count)	**790 C, 60s,Quench (55%γ, Point Count)**	**770 C, 60s, Quench (35%γ, Point Count)**	**860 C, 60s, Quench (55%γ, Point Count)**
Austenite (Martensite) %	34.03	50.54	30.2	57.5
RXD Ferrite, %	36.82	27.26	52.8	32.4
Non-RXD Ferrite, %	27.78	22.20	16.9	10.1
Normalized RXD Ferrite (RXD Ferrite/Total Ferrite) %	**56.9**	**55.11**	**75.7**	**76.2**

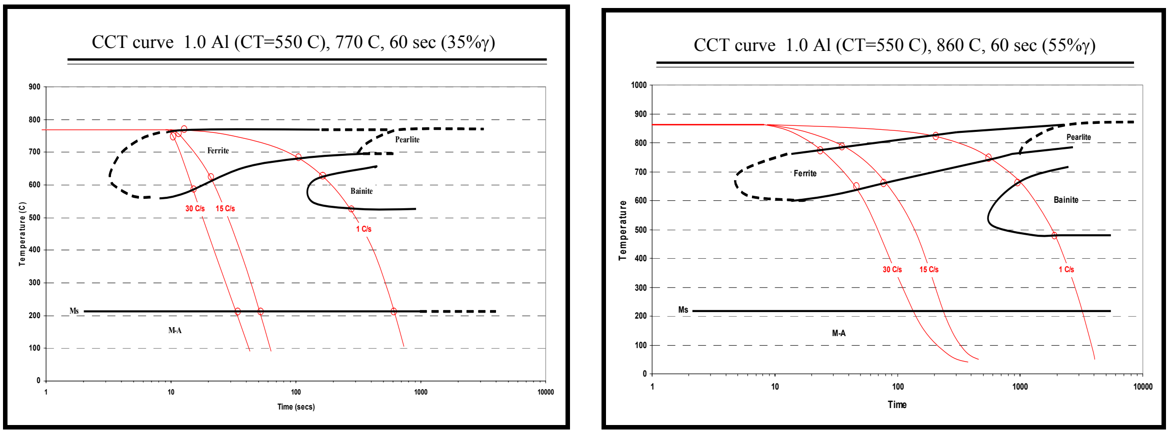
b) Cooling from T_{IA} to IHT

Figure 3a) and 3b) show the continuous cooling transformation diagrams (CCT) of the 0.05Al-550, for an initial austenite percent of 35%(lower T_{IA}) and 55%(higher T_{IA}), respectively. In the first case, it is observed that epitaxial or "new" ferrite is formed during cooling in the temperature range of 705°C and ceases at ~620°C at essentially all cooling rates. Ferritic bainite forms at a temperature close to 460°C at both 15 and 30°C/sec, and this start temperature increases to 540°C at slower cooling rates, 1°C/sec. Furthermore, granular bainite is also observed when cooling at 1 °C/sec. When cooling from an initial γ=55% condition, both the ferrite and bainitic ferrite start temperatures increased. Increasing the Al content to 1.0% eliminates the bainitic ferrite region, and only a "new" ferrite region is observed, regardless of prior coiling temperature. It appears that when a T_{IA}= 35%γ is used, the epitaxial or "new" ferrite formation immediately begins on cooling as observed in Figures 4a) and 4b). When a larger austenite percent is used, 55%γ, an undercooling is necessary before the formation of new ferrite can be observed, as shown in Figure 4b). Moreover, note that there is no pearlite formation even at a slow cooling rate of 1°C/sec, however, granular bainite can be observed at this slow cooling rate. Figure 5 shows SEM micrographs of the typical microstructures found in both 0.05Al-550 and 1.0Al-550 alloys at both intercritical annealing temperatures equal to an initial 35%γ.



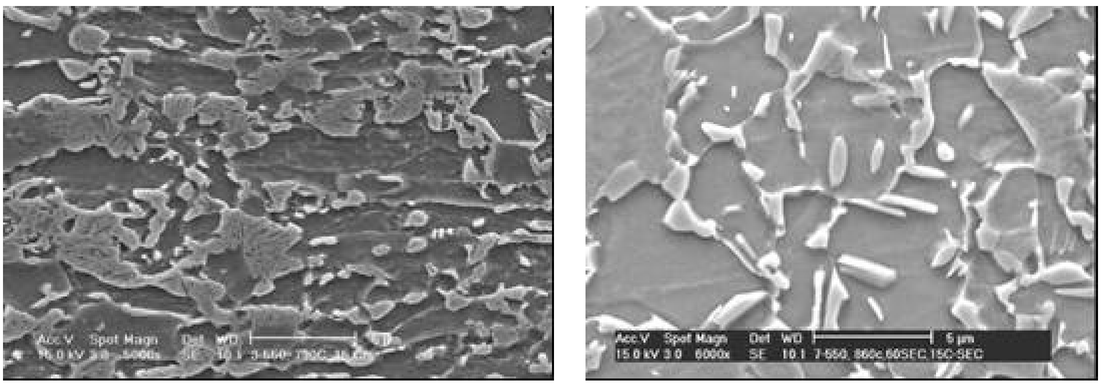
(a) (b)

Figure 3: CCT diagram from (a) T_{IA}=750°C (35%γ), and (b) T_{IA}=790°C (55%γ) in the 0.05Al-550 TRIP steel



(a) (b)

Figure 4: CCT diagram from (a) T_{IA}=770°C (35%γ), and (b) T_{IA}=860 C (55%γ) in the 1.0Al-550 TRIP steel



(a) 0.05Al-550 (b) 1.0Al-550

Figure 5 Typical SEM micrographs after cooling from T_{IA}=55%γ to room temperature at 15°C/sec. Specimens were tempered for 3 hrs at 200°C to develop martensite-austenite contrast. In the 1.0Al-550 alloy, no carbide structure was observed in the M-A particles after the tempering treatment, suggesting that these particles are γ_r.

It was observed that a large percent of the austenite formed during intercritical annealing transformed to ferrite during cooling from the intercritical annealing temperature to the isothermal

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holding temperature. The effect was observed by Optical Microscopy and EBSD-IQ analysis in the three steels studied under all circumstances and the total amount of transformed ferrite was found to be most influenced by both the Al content and the initial austenite percent at the intercritical annealing temperature (and therefore largely influenced by the carbon content of the austenite). Figure 6 clearly shows that a larger initial austenite volume fraction during intercritical annealing will result in a larger volume fraction of new ferrite after cooling to the IHT, and similarly, the 1.0Al alloy resulted in a larger volume fraction of new epitaxial ferrite during cooling.

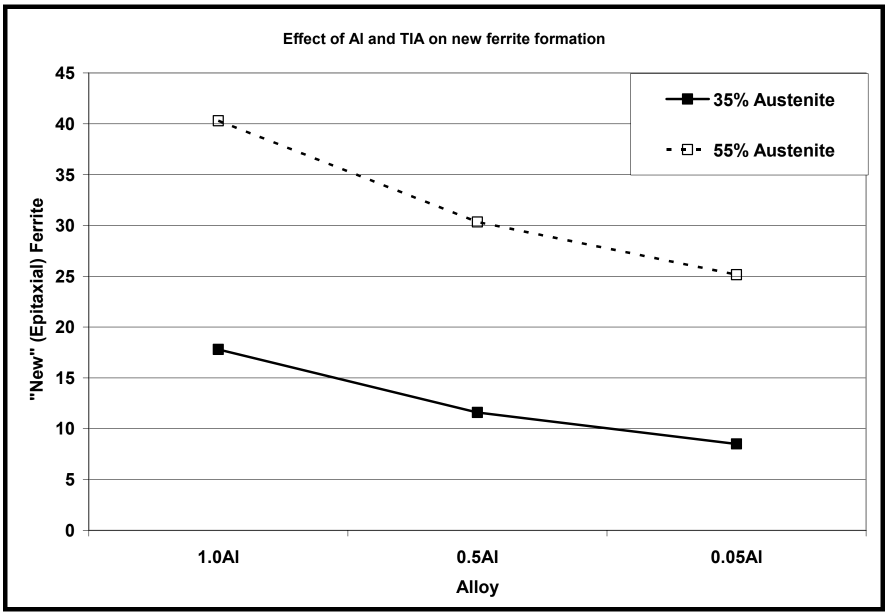

Figure 6: Effect of Al addition and intercritically annealed austenite on the volume fraction of new ferrite formed during cooling at 15°C/sec from T_{IA}= 35%γ and T_{IA}= 55%γ to IHT=450°C, and quench.

c) Isothermal Holding Temperature

The austenite decomposition during isothermal holding at 450°C was studied in the three Al bearing alloys, and it was observed that in all cases the volume fraction of austenite to bainite transformation during this holding period is relatively small, i.e., only about 2-14% after holding for two minutes, independent of initial volume fraction of austenite, cooling rate from the intercritical annealing temperature to the isothermal holding temperature (450°C) or prior coiling temperature conditions. These results are shown in Figure 7 for the 1.0Al-550 alloy, where the volume percent of the different phases is plotted at different time intervals during holding at 450°C. In this case, the annealing temperature corresponds to an initial austenite volume fraction=35%, then cooled at 5 and 15°C/sec to 450°C. It can be observed that the variation in the cooling rate has no significant effect on the total volume fraction of the final phases. However, it was observed that cooling at 5°C/sec resulted in a peak in the amount of retained austenite at short holding times, whereas cooling at 15°C/sec no peak is observed, at least during the time interval investigated, and the volume fraction of retained austenite increased with holding time. It can be observed that the amount of martensite gradually decreases and disappears shortly after 60 seconds. Similarly, the maximum volume fraction of bainite after 120 seconds is ~3%. An additional 2% is obtained during cooling from the IHT to RT (room temperature), as observed from the difference in the 2sec+AC and 2sec+quench specimens. A similar analysis is shown in Figure 7b), for an initial austenite volume fraction of 55% (860°C). Note that the maximum amount of bainite is approximately 1%. Also, very little martensite is observed. Furthermore, a similar analysis was undertaken in the 0.5Al-550 alloy intercritically annealed at 760°C (35%γ), Figure 8a). Note that the amount of retained austenite is less than that obtained in the 1.0Al-550 alloy, and the volume fraction of martensite is larger. Similarly, the maximum volume fraction of bainite in this 0.5Al-550 alloy is 6.1% and an additional 2.6% is obtained during cooling. Finally, Figure 8b shows a similar analysis for the 0.05Al-550 alloy intercritically annealed to obtain an initial 35%γ. After holding for 30 seconds at 450°C, the volume fraction of bainite reaches 10.3% and the volume fraction of austenite is only 2%. Holding for 120 seconds, increases the bainite volume fraction to 14% and the retained austenite volume fraction is increased to 4.1%.

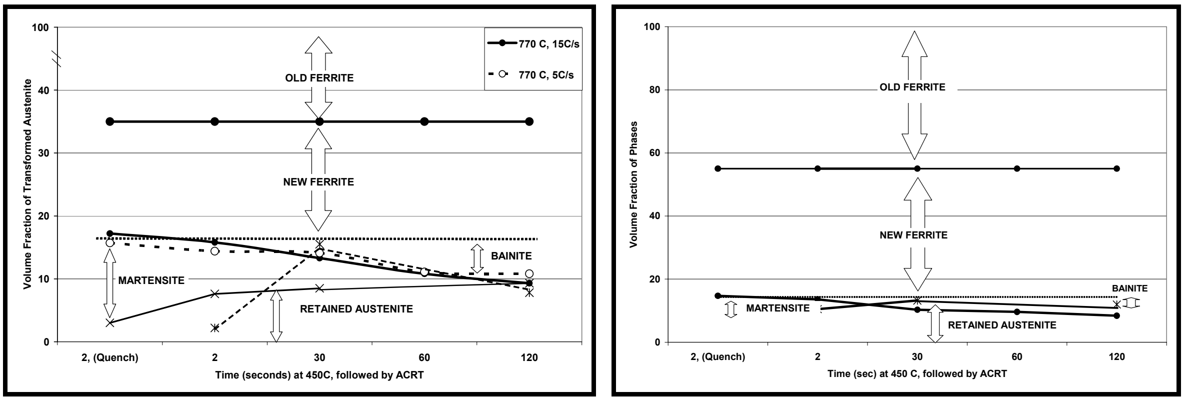

Figure 7: Austenite decomposition behavior during isothermal holding at 450°C after cooling from a) T_{IA}=35%γ, 1.0Al-550 TRIP steel, b) T_{IA}=55%γ, 1.0Al-550 TRIP steel

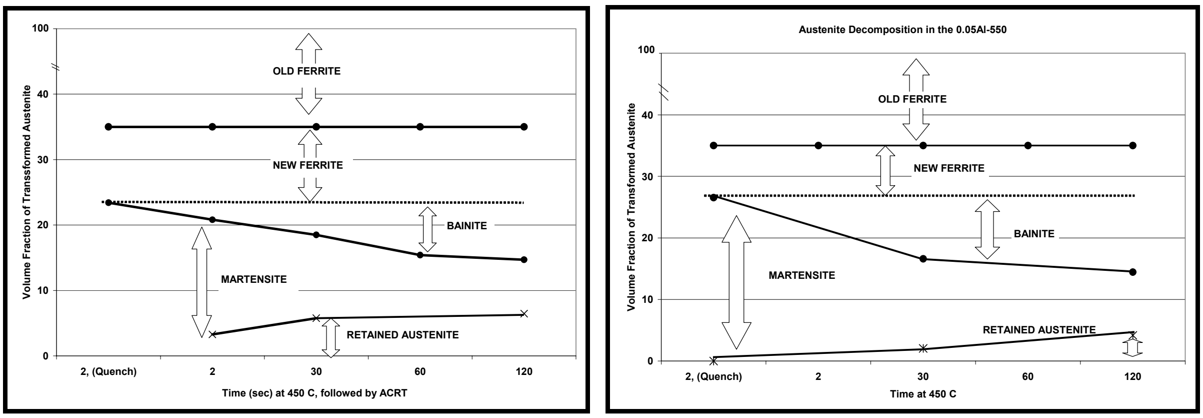

Figure 8: Austenite decomposition behavior during isothermal holding at 450°C after cooling from a) T_{IA}=35%γ, 0.5Al-550 TRIP steel, b) T_{IA}=35%γ, 0.05Al-550 TRIP steel

The extent to which the bainite reaction occurs during the isothermal holding region is strongly influenced by the Al content in the alloy. Figure 9 shows the dilatometric data obtained during the isothermal holding temperature, and it displays a number of important points for describing the metallurgical phenomena that occur during isothermal holding at 450°C. It can be observed that the reactions begin very rapidly through their first few seconds, and then the reactions slow and level off, having been mostly completed after about 60 seconds for T_{IBT} = 450°C in the 1.0Al and 0.5Al alloy, whereas in the 0.05Al alloy the reaction continues even after 300 seconds.

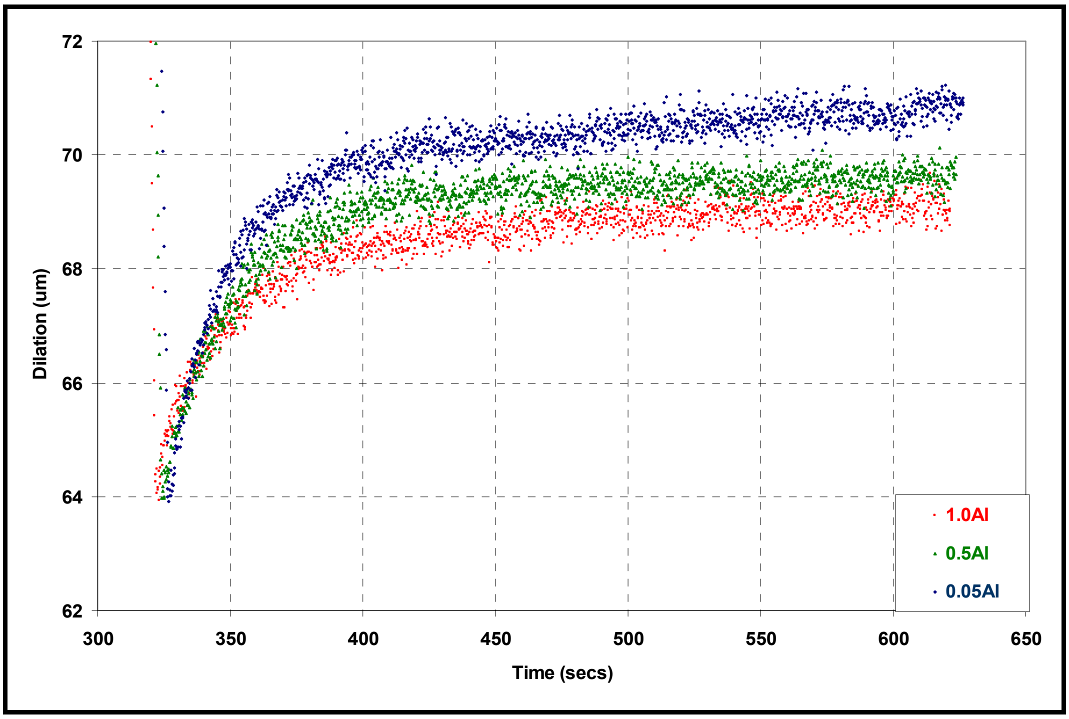

Figure 9: Dilatometry curves for the 0.05Al, 0.5Al and 1.0Al TRIP steels after intercritical annealing at γ=35%, IHT=450°C, and held for 5minutes.

It was observed that the UTS decreased with holding time during the IHT [9]. However, the tensile properties became nearly constant after just one minute of IHT time. This can be explained in terms of the leveling-off of the IBT dilatometry curves before the one minute mark is reached; that is, because the reaction is arrested shortly after a minute, and therefore, the result are very similar mechanical properties.

The observed large jump in mechanical properties between the specimens held for 5 seconds and the specimens held for 30 can be explained in terms of the rapid reaction at short IBT times shown in [9]. The specimen held for 5 seconds experienced only a small bainitic reaction whereas the specimen held for 30 seconds experienced most of the bainite reaction during the holding, in addition to the post-cooling stage.

Dilatometry showed that the first few seconds of the reaction are the most profound, so it comes as no surprise that even a short treatment in the bainitic temperature regime would cause a large difference in mechanical properties than, for example, the difference experienced the specimens held at 60 seconds and 120 seconds.

Furthermore, hardness (Vickers) measurements in the 1.0Al-550 steel agree with the shape of the dilatometry curve. During the first few seconds of isothermal holding, there is a clear change in hardness (independent of initial austenite percent), and after 30 seconds, the hardness values remain essentially constant. Obviously, the drop in hardness is associated with the decrease in M-A volume fraction, rather than the bainite formed during this holding period (which is less than 4%).

Despite the fact that the hardness values remain almost constant after holding for 30 seconds, the tensile strength and elongation values are observed to change with holding time. Given the relatively small magnitude of the bainite reaction in the 1.0Al alloy, equal to about 4% volume fraction, it is surprising to observe such changes in tensile strength and total elongation. Almost

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identical results were observed by Parish. As suggested in his research work [10], rather than austenite enrichment, a homogenization of carbon is taking place.

The cooling rate after holding at different time intervals during the isothermal holding at 450°C was varied and it was observed that the stability of the retained austenite changes as a function of the cooling rate after any given holding during the IHT. Figure 10 shows the effect of cooling rate after holding at 450°C on the amount of retained austenite for the 1.0Al-550 alloy. It can be observed that a systematic variation in the amount of retained austenite with varying cooling rates exists. Air cooling after holding at 450°C resulted in the largest amount of retained austenite, whereas quenching resulted in the smallest amount of retained austenite. Also, note that the percent of retained austenite increases with holding time, up to a maximum of 10.5% when air cooling is used, and up to 5% if quenching is used after IHT.
Also, it was observed that the prior coiling temperature has a significant effect on the retained austenite content. A prior coiling temperature of 700°C results in less than 2% retained austenite after holding for 30 seconds, and further holding decreases the volume fraction of retained austenite to zero.

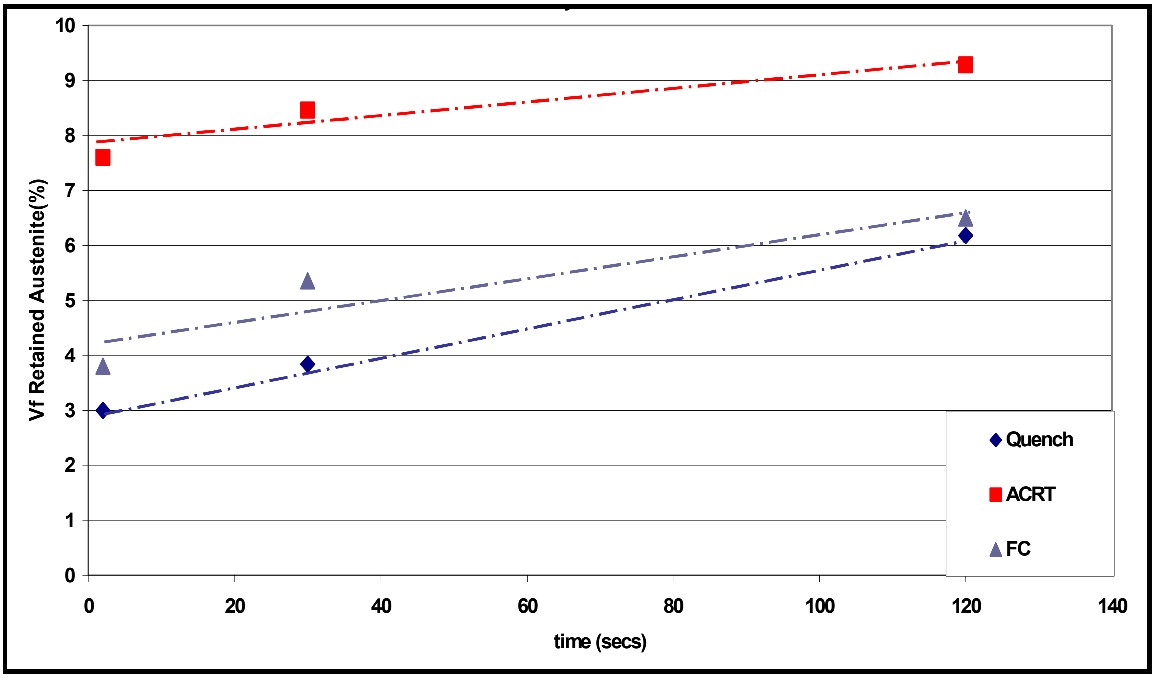

Figure 10: Effect of the cooling rate from IHT=450°C to RT after holding for various times on the volume fraction of retained austenite in the 1.0Al-550 TRIP steel

SUMMARY

The addition of Al is known to cause an increase in the activity of carbon in the austenite, and therefore the driving force for diffusion of carbon away from the interface is increased. It appears that the critical Al content for a significant reduction of carbon segregation towards this interface is about 1.0 wt%, and in consequence, the mobility of the grain boundaries increases. This would in fact explain why the addition of 1.0%Al results in much faster rates of recrystallization. Moreover, this also explains why the variation in coiling temperature has essentially no effect in the type of ferrite (due to the lack of carbon build up at the α/γ when lowering the transformation temperature) and similarly, the reduced effect of carbon decorating the Mn banding in the 1.0Al steel.

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The large influence of the intercritical annealing temperature on the volume fraction of new ferrite can also be explained in terms of the austenite hardenability A reduction in the intercritical annealing temperature will result in a smaller fraction of intercritical austenite, with higher carbon content and therefore higher hardenability. In consequence, this will lead to a slower epitaxial ferrite reaction. As the epitaxial ferrite reaction will be diffusion controlled, a higher carbon concentration will require more time to deplete the transformation-front diffusionally.

Finally, it was observed that the amount of new ferrite formed during cooling appears to depend on the alloy content. In the 0.05Al alloy, cooling from 750 C (35%γ) to 450 C results in ~8.5% new ferrite whereas in the 1.0Al alloy the new ferrite formed is ~18%. This supports the suggestion that the addition of 1.0%Al to this alloy system increases the mobility of grain boundaries by reducing the carbon segregation to the grain boundaries as discussed previously.

CONCLUSIONS

It was established that it is possible to obtain TRIP steels with excellent mechanical properties under conventional CGL processing parameters by using Al, Nb-Mo additions. It was observed that the best TRIP steel was the 1.0Al-550 alloy, which achieved tensile strengths of 800MPa and elongations larger than 25%. The following important conclusions can be made:

a) STAGE I. Intercritical Annealing: It was observed that Al additions greatly affect the Fe- C phase diagram, expanding the α+γ region, and increasing both the Ac1 and Ac3 temperature. Additions of 1.0Al wt% increased the Ac3 temperature close to 1000 C. Also, it was observed that the rate of ferrite recrystallization was substantially increased and fully recrystallized grains in the 1.0Al alloy were observed at temperatures well below intercritical annealing temperatures equal to 35%γ or higher. In addition, Al additions >0.5%wt eliminated the effect of prior coiling conditions on the volume fraction of austenite during intercritical annealing and on the recrystallization of ferrite.

b) STAGE II. Cooling from TIA to IHT: It was observed that the volume fraction of "new" epitaxial ferrite is strongly influenced by both the Al additions and the intercritical annealing temperature. More "new" epitaxial ferrite is formed with increasing Al additions and higher 172intercritical annealing temperatures. Also, it was observed that increasing the Al content to 1.0%wt eliminated the bainite curve in the CCT diagrams, and retained austenite was observed rather than martensite. No significant effect of prior coiling conditions on the volume fraction of new epitaxial ferrite was observed.

c) STAGE III. Isothermal Holding Temperature: It was observed that the bainite reaction does not occur extensively in Al added Nb-Mo TRIP steels. Only about 4% was present and only after holding for 2 minutes in the IHT=450°C. Also, it was clearly observed that the stability of the retained austenite is not associated with the bainite formation. Retained austenite particles were observed along grain boundaries and inside ferrite grains. Furthermore, stable retained austenite was obtained at short holding times, and therefore long isothermal holding times are unnecessary. In addition, the cooling rate after IHT can have a significant effect on the amount and stability of the retained austenite. It was observed that the optimum cooling rate is air cooling. Faster cooling rates result in a larger amount of martensite, whereas slower cooling rates result in carbon depletion towards the grain boundaries (as observed from the yield point elongation in the tensile curves). Finally, essentially no retained austenite was observed when the specimens where prior-coiled at 700 °C.

d) Mechanical Properties: A lower initial austenite percent during intercritical annealing results in better tensile and elongation properties. Tensile strength decreased and elongation increased with holding time during the IHT. The goal of UTS=800 MPa and %El=25-30% was achieved with the 1.0Al-550 TRIP steel using heat cycles normally observed in a CGL, eliminating the necessity of long IBT holding sections.

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