

VANADIUM AND ALUMINUM COMPETITION FOR NITROGEN IN MICROALLOYED FORGING STEELS

L.M. ROTHLEUTNER¹ AND C.J. VAN TYNE²

¹Graduate Research Assistant and FIERF Forging Fellow, Dept. of Metallurgical and Materials Engineering, Colorado School of Mines, Golden, CO 80401 USA

²FIERF Professor, Dept. of Metallurgical and Materials Engineering, Colorado School of Mines, Golden, CO 80401 USA

ABSTRACT

When aluminum nitrides form in a microalloyed steel that contains vanadium, the amount of nitrogen available for precipitation with the vanadium is decreased, which causes a potential decrease in the precipitation strengthening. In this study, the interaction of vanadium, aluminum, and nitrogen was investigated using a commercial vanadium-microalloyed medium-carbon bar steel (38MnSiVS5 (modified AISI 15V41)) with three aluminum levels. Laboratory thermal simulations with varied reheat temperatures (1050 to 1250 °C – 1925 to 2275 °F) and hold times (300 to 2700 s – 5 to 45 min) were used to study the interaction of the various components. Microstructures and properties were measured using quantitative metallography, microhardness, and quantitative chemical analysis of electrolytically extracted precipitates. The extent of precipitation strengthening, as measured by hardness, was relatively constant for all aluminum levels at and above 1150 °C (2100 °F) independent of hold time. Average hardness, pearlite fraction, and austenite grain size decreased with increasing aluminum content at the two lowest temperatures examined, 1050 (1925) and 1100 °C (2010 °F). It was also found that the amount of vanadium precipitate significantly decreased with an increase in aluminum content.

INTRODUCTION

A variety of hot forged steel products are presently produced using microalloyed (MA) steels, which are directly cooled from the press or hammer. For certain applications, MA steels can provide an overall lower-cost alternative to quenched and tempered (QT) steels. Cost savings come primarily from the elimination of post-forging heat treatments. Typical forged components of MA steels are crankshafts, connecting rods, spindles, and wheel hubs (1).

The major microalloyed element in forging steels is vanadium (V), which is used in small amounts, often on the order of 0.1 wt pct or less. These forging steels with V are typically medium-carbon (0.30 to 0.50 wt pct) ferrite-pearlite or bainitic steels (2). The V additions provide a strength increase because of precipitation hardening. The V precipitates typically increase strength by 50 to 200 MPa (7 to 30 ksi). These higher increases in strength are achieved through precipitating very low-volume fractions (typically 10^{-4} to 10^{-3}) of very fine (typically <20 nm) incoherent vanadium nitride (VN) or vanadium carbo-nitride (V(C,N)) precipitates (3).

The objective of the present study was to examine the influence of aluminum (Al) on V microalloying additions during the heating cycles during a forging process. The issue is whether or not Al reacts with nitrogen in the steel to produce aluminum nitride (AlN) precipitates. When AlN precipitates form in the steel at high temperatures, the available amount of nitrogen for precipitation with V is decreased, which causes a decrease in the potential for precipitation strengthening. Aluminum is often used to produce a fine-grained steel product, since aluminum nitrides can refine the austenite grain structure through grain boundary pinning (1).

The V additions usually precipitate as vanadium carbo-nitrides (V(C,N)) during the austenite decomposition. Since the formation of AlN and V(C,N) can occur simultaneously, a competition for nitrogen (N) ensues. Variations in forging parameters, such as forging temperature and time at temperature prior to forging, as well as the amount of deformation, can influence the balance between AlN and V(C,N) precipitates. The focus of this paper is to examine the heating cycles for a forging process and to determine the influence that Al has on the subsequent V(C,N) precipitation.

MATERIAL

A medium-carbon V microalloyed steel—38MnSiVS5 (modified AISI 15V41)—with three separate Al additions was studied. Table 1 shows the composition of this steel. The steel was continuously cast from a single heat with Al additions of 0.006, 0.020, and 0.031 wt. pct being made in the mold by wire feed. A reduction ratio of 4.3:1 was used to roll the billet to 117.5mm (4-5/8 in) round bar. Hereafter the steels will be designated as 6Al, 20Al, and 31Al, which refer to their respective Al content.

Table 1 – Chemical Composition (wt. pct) of the Modified SAE 15V41 Base Steel

C	Mn	Si	Cr	Ni	Mo	V	Al	N	S	P	Cu
0.37	1.36	0.63	0.13	0.08	0.02	0.088	var.	0.0153	0.064	0.010	0.18

EXPERIMENTAL PROCEDURES

A thorough analysis of the as-received steel bars was performed to understand variations in microstructure, chemistry, and properties so that specimens could be machined from a region in the bar that was the most consistent. Microstructural, chemical, and hardness characteristics of each alloy were coupled with appropriate statistical analyses. It was found that the mid-radius of the bar gave the most consistent microstructure, properties, and chemistry. Details about these analyses of the as-received bars can be found in the proceedings of the previous Forging Technical Conference (4).

As a laboratory simulation of the thermal cycle that steel sees during hot forging, the Gleeble® 1500 system at Colorado School of Mines was used to heat, hold, and then cool each specimen. The heating was chosen to simulate an industrial induction heating process, the hold times simulated both induction heating and gas fired heating, and the cooling was chosen to simulate the air cooling of a 76 mm (3 in) diameter bar.

A two-step heating schedule of 20 °C/s (36 °F/s) to 750 °C (1382 °F) followed by a 4 °C/s (7.2 °F/s) to the holding temperature was used. The change in heating rate at 750 °C (1382 °F) was intended to mimic the decreased efficiency of induction heating that occurs above the Curie temperature (i.e. the magnetic transition) of ferrite (770 °C/1418 °F).

The holding step was performed at each of five different temperatures for three different times. The holding temperatures were 1050, 1100, 1150, 1200, and 1250 °C (1922, 2012, 2102, 2192, and 2282 °F). The maximum holding temperature of 1250 °C (2282 °F) was selected since it is the recommended forging temperature for AISI 1541 (1245 °C/2273 °F). The remaining four temperatures were chosen to precipitate different amounts of AlN as determined by solubility product calculations. Durations of 5, 15, and 45 minutes at the holding temperature were used to account for both the shorter holding time of induction heating and the longer holding times associated with gas-fired furnaces.

The cooling step consisted of two cooling rates. The first part of the cooling step was a variable cooling rate from the holding temperature to 750 °C (1382 °F) in 6 min. This cooling results in an initial cooling rate that varies from 0.83 to 1.39 °C/s (1.5 to 2.5 °F/s). The cooling rate was then reduced to 0.25 °C/s (0.45 °F/s) until the specimen reached 500 °C (932 °F), after which the specimens were allowed to freely cool to room temperature.

After the heat treatment, all specimens were analyzed microscopically. Features such as ferrite fraction, pearlite fraction, and austenite grain size were quantified.

Hardness tests were performed on each sample with a Vickers microhardness test unit. Twenty-five hardness tests were made on each specimen.

Quantification of Al and V dissolved in the steel, as well as in the precipitate formed, was performed by electrolytically extracting the precipitates. During the extraction process, the ferrite was selectively dissolved electrochemically (electrochemical dissolution) using a non-aqueous electrolyte. The extracted precipitates were filtered from the electrolyte and digested in strong acids. Both the extraction solution, which contained the dissolved elements and the digested precipitates, were analyzed through inductively coupled plasma mass spectrometry (ICP-MS).

RESULTS AND DISCUSSION

The usual microstructure of a 15V41 steel forging component would be a mixture of ferrite and pearlite. The amount of pearlite is directly proportional to the strength of the component. The amount of pearlite can vary depending on the forging temperature and the cooling conditions. In the laboratory tests, the rate of cooling was kept constant, so any variation of pearlite fraction is primarily due to the holding (i.e. forging) temperature.

Figure 1 shows pearlite fraction versus hardness for the laboratory simulations. In Figure 1a, a linear relationship is seen between pearlite fraction and hardness for the three higher temperatures (1150, 1200, and 1250 °C). The hardness value, which is directly related to strength, depends both on the pearlite fraction and the amount of precipitation in the steel. The observed linear trend indicates that at 1150 °C (2102 °F)

and above, the hardness is directly dependent on the pearlite fraction and the strength increase due to precipitation of V(C,N) would be constant.

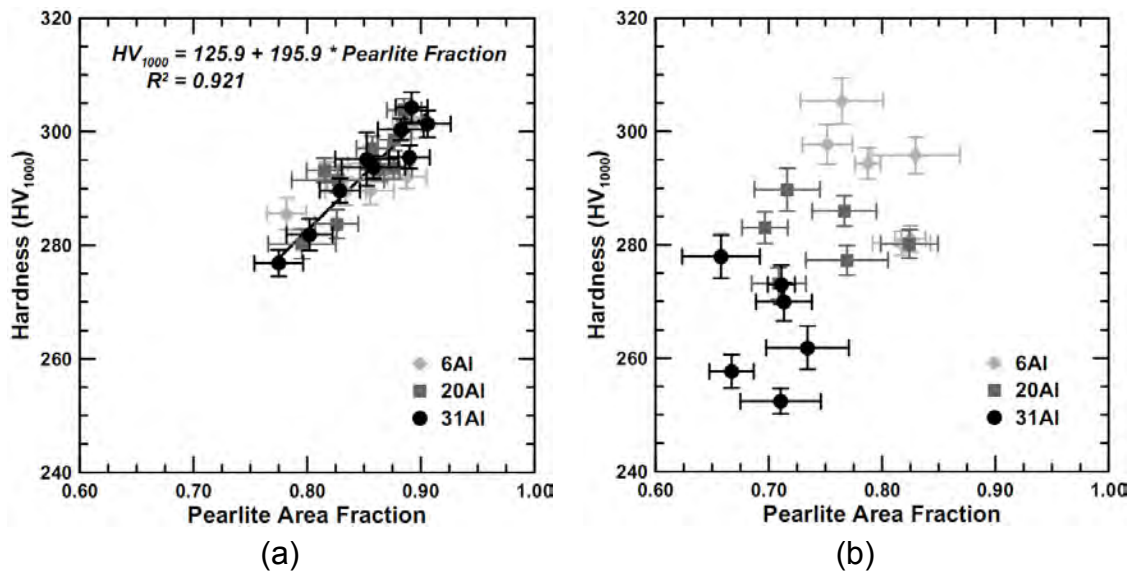


Figure 1 Pearlite area fraction versus Vickers microhardness for (a) 1150, 1200, and 1250 °C, as well as for (b) 1050 and 1100 °C thermal simulations. Error bars represent 95 pct confidence intervals for the mean.

At the two lowest temperatures (1050 and 1100 °C/1922 and 2012 °F), no obvious trend was observed between pearlite area fraction and hardness; however, the hardness appears to decrease with increasing Al content. This dependence of hardness on Al content at 1050 and 1100 °C suggests that the increased AlN precipitation at these lower temperatures influences either V(C,N) precipitation strengthening and/or the austenite transformation behavior during cooling. An increase in the amount of AlN precipitation would reduce the amount of N available for V(C,N) precipitation therefore reducing the precipitation strengthening potential of the V(C,N). Austenite transformation behavior can be influenced by an increase in AlN precipitation which would pin the grain boundaries and reduce the austenite grain size.

Figure 2 shows the austenite grain size at 1050, 1100, and 1150 °C for both the 6Al and 31Al alloys as a function of time. The 31Al alloy exhibits a significantly smaller grain size at 1050 and 1100 °C than the 6Al alloy under the same conditions. Of the nine lab simulations examined for austenite grain size, the 31Al alloy held for 45 min at 1150 °C was the only condition that did not exhibit significantly inhibited grain growth when compared to the 6Al alloy. The decrease in austenite grain size with higher Al content is a clear indication that AlN precipitation is occurring at lower temperatures of 1050 and 1100 °C. With an increase in AlN, it would be reasonable to expect that the amount of V(C,N) would decrease. The electrolytic extraction experiments were designed to see if this was indeed the case.

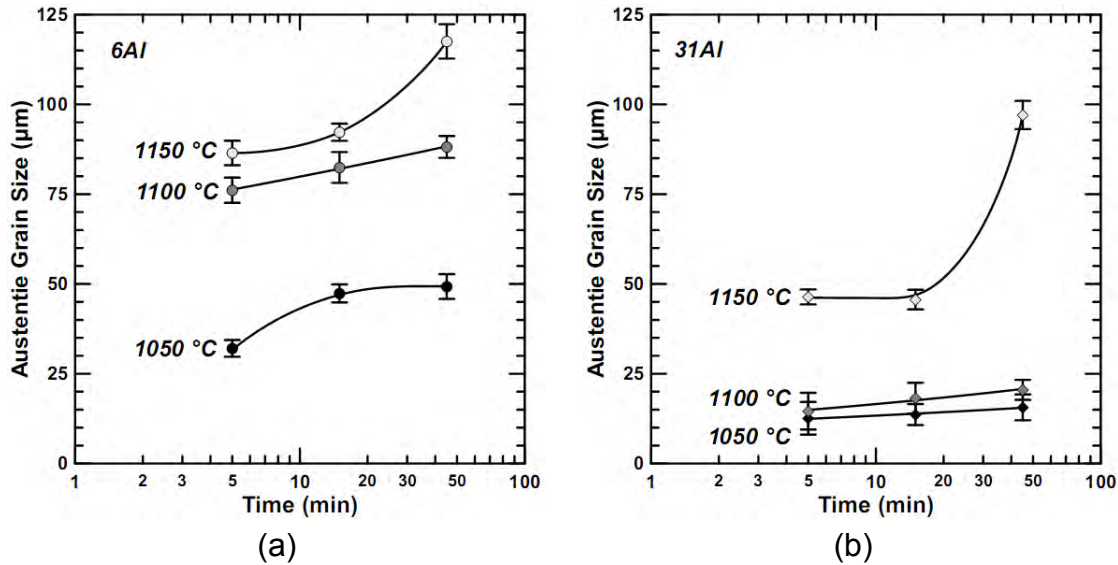


Figure 2 Austenite grain size as a function of time for 1050, 1100, and 1150 °C hold temperatures for the (a) 6Al and (b) 31Al alloys. Error bars represent 95 pct confidence intervals for the mean.

The electrolytic extraction results indicated that all of the heated and cooled specimens had a significantly larger amount of Al precipitates and smaller amounts of V precipitates when compared to the as-received condition. These changes clearly indicate that during the forging cycle, Al reacts with N, which decreases the strengthening potential for the V precipitates.

For the laboratory simulations, the amount of V precipitated in the 6Al alloy was consistent for all conditions (approximately 70 pct); while the 31Al alloy had less V precipitated than the 6Al for all conditions. The specimens held at 1050 and 1100 °C for 15 min exhibited essentially equal amounts of Al and V precipitates. The specimens held for 45 min at 1050, 1100, and 1150 °C showed a maximum Al precipitation of 75 pct at 1050 °C. The amount of AlN precipitation steadily decreased as the temperature increased. The amount of Al precipitated under these conditions is inversely related to the amount of V precipitated. Figure 3 shows the amount of V precipitated as a function of the amount of Al precipitated for 6Al and 31Al specimens examined. The linear behavior indicates a clear competition for N between AlN and V(C,N) precipitation in the 6Al and 31Al alloys.

The result of the excess Al in the steel will provide a component with a finer grain size, but the strengthening potential from the V will be diminished. As in many metallurgical situations, appropriate and knowledgeable decisions must be made to balance these conflicting mechanisms.

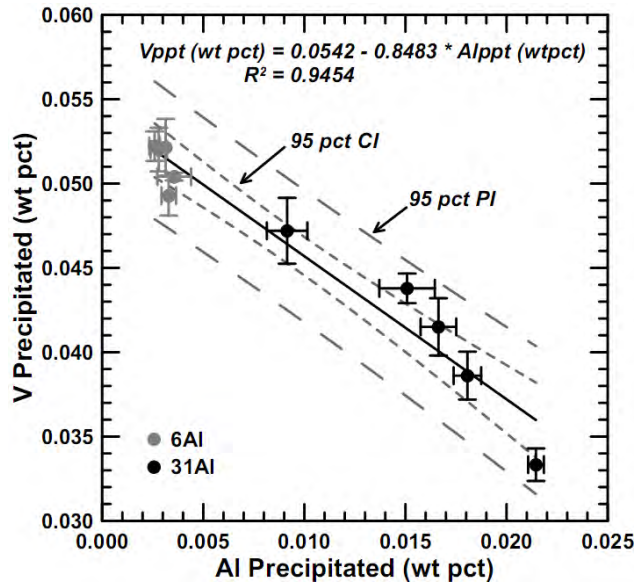


Figure 3 Amount of V precipitated as a function of Al precipitated for the 6Al and 31Al alloys for select thermal simulations (min-°C): 15-1050, 15-1100, 45-1050, 45-1100, and 45-1150. Error bars represent plus or minus one standard deviation of the mean. Confidence and prediction intervals for the linear model at the 95 pct level are shown.

SUMMARY AND CONCLUSIONS

A linear relationship was observed between pearlite fraction and hardness at the three higher temperatures examined (1150, 1200, and 1250 °C/ 2102, 2192, and 2282 °F) for all hold times and at all Al levels. The hardness variation is primarily due to the pearlite fraction and the strength increase from precipitation of V(C,N) is constant. A trend of lower hardness for higher aluminum content was somewhat observable at the two lowest temperatures (1050 and 1100 °C/1922 and 2012 °F). These results indicate that although AlN decreases the austenite grain size, it consumes N, which results in a lesser precipitation strengthening from the V(C,N).

All laboratory simulations of the forging thermal cycle had higher amounts of Al precipitation as compared to the as-received steels. The simulations with 15 and 45 min hold times at 1050 and 1100 °C, as well as with a 45 min hold at 1150 °C for both the 6Al and 31Al alloys, were evaluated using the electrolytic extraction method. The results showed that the 31Al alloy had less V precipitated than the 6Al for all conditions. Simulations that had a larger amount of Al precipitated had a smaller amount of precipitated V, which clearly demonstrates the competition for N between AlN and V(C,N) precipitation.

REFERENCES

- (1) G. Krauss, *Steels: Processing, Structure, and Performance*, Materials Park, OH: ASM International, 2005.
- (2) C.J. Van Tyne, "Forging of Carbon and Alloy Steels," in *ASM Handbook Volume 15: Metalworking: Bulk Forming*, Materials Park, OH: ASM International, 2005, pp. 241-260.
- (3) W. Morrison, "Overview of Microalloying in Steel," in *The Use of Vanadium in Steel*, Guilin, China, 2000, pp. 25-35.
- (4) L.M. Rothleutner, A.S. Hering and C.J. Van Tyne, "Property and Microstructure Variation in Forging Bar Steels", in *CD Proceedings of 28th Forging Industry Technical Conference*, Forging Industry Education and Research Foundation, Cleveland, OH, USA, 2011.

ACKNOWLEDGEMENTS

Support for this work by the Advanced Steel Processing and Products Research Center at the Colorado School of Mines and by FIERF in the form of 2010-2011 FIERF fellowship for L.M. Rothleutner is gratefully acknowledged.