

Compositional Sensitivity of High Mn, High Al Lightweight Steels

**Katherine Sebeck, PhD¹, Ian Toppler¹, Krista Limmer, PhD², Daniel Field, PhD²,
Daniel Wagner¹, Alyssa Gafner¹**

¹Materials Division, US Army CCDC Ground Vehicle System Center, Warren, MI

²US Army CCDC Army Research Lab, Aberdeen, MD

ABSTRACT

The family of lightweight high Mn, high Al steels (FeMnAl) exhibit lower density (6.5-7.2 g/cm³) than traditional military steels (7.9 g/cm³). These alloys are precipitation hardened, with κ -carbide dominating hardening performance. This carbide has an E₂₁ perovskite structure with a nominal composition of (Fe,Mn)₃AlC. In the literature, a number of studies have examined the sensitivity of mechanical properties to changing a single element in the composition. However, the covariance of the major elements has not been systematically explored. In this study, a series of small ingots were prepared according to a two-factor design of experiments, in addition to analysis of previously generated compositions. Methods of measuring alloy composition will be discussed, along with aging kinetics and key mechanical properties.

Citation: K. Sebeck, I. Toppler, K. Limmer, D. Field, D. Wagner, A. Gafner, "Compositional Sensitivity of High Mn, High Al Steels", In *Proceedings of the Ground Vehicle Systems Engineering and Technology Symposium (GVSETS)*, NDIA, Novi, MI, Aug. 13-15, 2019.

1. INTRODUCTION

The increasing weight of Army ground vehicle systems has been a challenge for both system sustainment and new system design. [1] The FeMnAl steel alloy system has been investigated over the last several years as a potential lightweight material for these applications. [2] [3] This alloy is being developed to meet the same performance requirements of the existing armor steel, at a reduced density, with an emphasis on development of appropriate standards documents. In materials standards, in addition to the expected performance

based criterion, limits are established for compositional variation beyond the initial declaration of chemistry. The role of each alloying element and acceptable ranges for variation have been well established for traditional steels. However, for this new family of lightweight high strength steels, such thresholds and roles have not been definitively established.

Prior work in this family has focused on variation of a single element at a time. [4] [5] [6] Impact toughness is also a rarely reported metric, but critical to meeting performance specifications. Kalashnikov et. al. created a series of rods, examining Al from 3-10%, C from 0.85-1% and Mn of 24-34%, focusing on the δ -ferrite fraction

and U-notch impact toughness. [7]. However, this work assumed these alloying elements could be considered as independent variables. Bartlett examined the effect of secondary elements, silicon and phosphorous, within the cast form of the alloy. [6] While Si does not directly participate in the κ -carbide, it was found to increase the activity of carbon in solution, increasing the partitioning of the carbon.

The κ -carbide precipitates by a combination of spinodal decomposition and short-range ordering reactions, with a nominal composition of $(Fe,Mn)_3AlC$. This reaction will depend on the activity and availability of the constituents in solution. The ordered carbide is a soft dislocation barrier, and if present on grain boundaries, may lead to transgranular cleavage. Based on this precipitation path, we are explicitly exploring the covariance of the major elements of the carbide.

2. METHODOLOGY

This study examined two data collections: first, all data collected from preliminary industrial development of a single target composition was statistically analyzed. Due to the production challenges with this high alloy content, there is variation between batches of material.

Additionally, a two-level, 3 factor design of experiments (DoE) was applied, testing the contributions of Mn, Al, C and the ratios thereof, with the current composition defining the upper level for each elements. The objective of this DoE is to maximize the combination of Charpy V-notch (CVN) impact strength and Brinell hardness number (BHN) as a function of composition. The impact strength was measured by ASTM E23, while hardness were measured according to ASTM E18 for Rockwell C, and then converted to Brinell hardness values. [8] [9] Hardness values were measured through thickness to verify homogeneity. In addition to these compositions, all previously produced plates were statistically analyzed for any correlations. The target compositions of the DoE are listed in Table 1.

Table 1. Target compositions for design of experiments

Composition	Mn (wt%)	Al (wt %)	C (wt %)	Fe (wt %)
A	25.0	8.0	0.80	64.7
B	25.0	9.0	0.80	63.7
C	25.0	8.0	0.90	64.6
D	25.0	9.0	0.90	63.6
E	29.0	8.0	0.80	60.7
F	29.0	9.0	0.80	59.7
G	29.0	8.0	0.90	60.6
H	29.0	9.0	0.90	59.6

All compositions include a target of 1.0 wt% Si and 0.5 wt% Mo, with the balance as iron. This results in a range of Fe from 59.6 wt% to 64.7 wt%.

Two different vendors were selected to allow for overlap, in the likely event of casting defects, rolling failures, or deviations from the compositional target. It was anticipated that several compositions would only be completed by one vendor within the scope of this report. Throughout this document, Table 1 compositions will be referred to as Vendor-Composition, e.g., A-D would be composition D prepared by Vendor A.

Ingots made by Vendor A were produced using vacuum induction melting, in a 5”x5”x12” mold. These castings were then cut to have parallel faces, and hot rolled by Vendor A. Ingots made by Vendor B were produced using a Y-block sand mold. This mold was optimized to minimize inclusions and other defects. After casting, these pieces were sectioned to 2.5”x5”x8” blocks, before rolling at Vendor A. All plates were rolled to 0.5” thickness. These plates were then sectioned for aging studies and mechanical testing. Prior studies of the aging response of the initial composition were leveraged for the current work. [10]

Following production of these ingots, a series of compositional tests using three different methods were performed: inductively coupled plasma spectroscopy (ICP), x-ray fluorescence (XRF) and optical emission spectroscopy (OES). It was determined from these tests that OES with appropriate commercial standards was a viable option.

3. RESULTS

In previous efforts with a limited range of target compositions, a strong difference in Charpy impact toughness was observed between the softer, solution treated (STQ) condition, versus the harder, aged condition. A classic ductile failure mode is seen in the STQ sample, whereas the aged sample shows clear brittle intergranular failure, as shown in Figure 1.



Figure 1. Microstructure at Charpy fracture surface in the (upper) solution treated condition and (lower) aged condition

As the hardness increased, the impact toughness dropped off rapidly. To understand how best to mitigate this behavior, all of the available composition, hardness and toughness data was compiled.

3.1. Preliminary Correlation Analysis

Data from 26 plates of various compositions and aging histories was compiled. Compositions represented a relatively narrow target range. Manganese ranged from 29.7-30.2 weight percent, Al ranged from 8.8-9.1 wt%, C ranged from 0.9-

1.07 wt%, Mo ranged 0.46-0.7 w% and Si ranged from 0.8-1.0 wt%. The Pearson product-moment correlation coefficients were determined using the `numpy.corrcoef` function. This statistical test effectively describes the linearity of the relationship between values. An absolute value over 0.5 is considered a strong association, while a value of -0.3 to 0.3 is considered weak to no meaningful association.

Table 2. Correlation coefficients for impact toughness and hardness in FeMnAl plates

Factor	Correlation to CVN	Correlation to Hardness
CVN	1	-0.819
Hardness	-0.819	1
Aging Temp	-0.634	0.457
Aging Time	-0.392	0.474
Mn	0.196	-0.115
Al	0.015	0.056
C	0.243	-0.226
Mo	-0.244	0.276
Si	-0.163	0.164

While as expected, the strongest correlation is a negative correlation between hardness and impact toughness. There is also a medium dependence on the aging behavior, particularly temperature. Compositionally, the results are less significant or predictable. Aluminum had no meaningful correlation to CVN and hardness, while the strongest compositional correlation was molybdenum, followed by carbon. However, only a narrow compositional window was explored in this analysis. This limited sensitivity to small changes is promising for production consistency.

3.2. Compositional Variations

Due to the challenges of high alloy steel production, the actual composition of the plate typically deviated from the target composition. Additionally due, to manufacturing challenges, two plates were not completed in time for inclusion into

this effort. Manganese varied from 24.3-25.9 wt% for a target of 25 wt%, and 27.9-30.1 wt% with a target of 29 wt%. Aluminum ranged from 7.8-8.3 wt% and 8.8-9.2 wt %. Carbon ranged from 0.73-0.83 wt% for a target of 0.8wt% and 0.86->1.0wt% for a target of 0.9 wt%. It is clear from these numbers that further refinement of process controls is needed for this alloy family. In particular, it seemed challenging to precisely target the desired amount of manganese at higher target levels.

3.3. Design of Experiment: Hardness and Microstructure

For each composition, an aging study was conducted. Based on previous results, a single temperature and time for solution treating was chosen. Three different aging temperatures of 500°C, 550°C and 600°C were used. In general, three aging groups were found based on the Mn and Al levels: fast & hard (low Mn), intermediate (high Mn, high Al), and soft & slow (high Mn, low Al). When the Mn content was lower, a lower C content also seems to improve aging response. For example, B-G and B-D, despite their matching carbon contents (0.91 wt%) show drastically different aging behavior.

Given a 0.5” thick plate, a target hardness range of 340-390 BHN was used to evaluate these plates. Even after 100 hours, some plates never achieved the minimum hardness requirement, which were considered part of the soft & slow category. A subset of the aging curves are presented below in Figure 2.

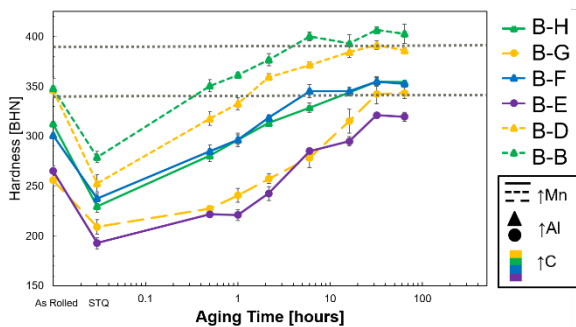


Figure 2. Example aging curves for a subset of plates showing the three regimes of aging

The two lowest Mn samples had the most rapid hardening behavior, while the two lowest aging rates were at the high Mn target, but low Al target. Plate B-E and B-G were did not achieve the target hardness in an industrially achievable time frame.

Compositions with an elevated Al content were more likely to have residual ferrite stringers after solution treatment, as seen in B-D, B-F, and B-H in Figure 3. These manifest in the scanning electron microscopy images as strong horizontal lines.

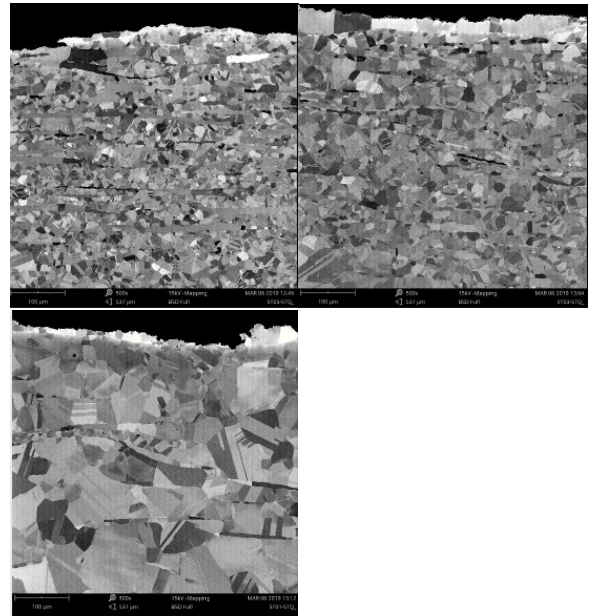


Figure 3. Surface microstructure following solution treatment of (upper left) B-D, (upper right) B-F, and (lower) B-H. 100 micron scale bar

There are also distinct differences in grain size, despite a consistent thermal history for all three samples. A more bimodal grain size distribution is seen in B-G and B-H below in Figure 4.

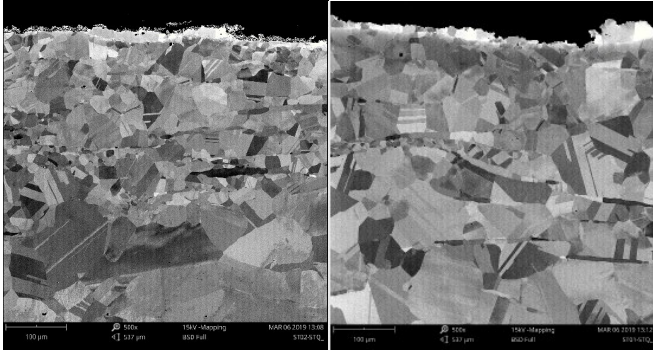


Figure 4. Surface microstructure following solution treatment of (left) B-G and (right) B-H. 100 micron scale bar.

Both also shown evidence of ferrite grains on the surface, which may be due to decarburization, as both are high Mn, high C compositions. However, these two compositions showed dramatically different aging behavior, with B-H reaching target hardness quite quickly, while B-G barely reached minimum hardness after 30+ hours.

3.4. Design of Experiment: Charpy Impact Testing

Several compositions were tested in the solution treated and water quenched, prior to aging heat treatment. Aging studies are also in progress for all samples. For those compositions that have successfully achieved the target hardness, an optimized aging strategy was determined, and a single heat treatment was then prepared for Charpy V Notch testing in the T-L orientation. Due to delays in material production, this work is ongoing.

A hardness target of ~350 BHN was chosen to safely be above the minimum threshold, but maximize expected impact toughness. Due to time restrictions, compositions falling in the “fast and hard” group were prioritized. This data is summarized in Table 5.

Table 3. Summarized Charpy V Notch and Hardness Data

ID	Condition	Hardness, BHN	CVN, ft-lbs @-40°C
B-B	Aged	352	8.49 ± 0.27
B-D	Aged	352	6.78 ± 0.33
B-B	STQ	271	34 ± 2
B-D	STQ	290	33 ± 2
B-E	STQ	268	93 ± 6
B-F	STQ	257	44 ± 3
B-G	STQ	253	163 ± 19
B-H	STQ	305	44 ± 8

For a hardness range of 250-270 BHN, the target impact toughness is 60 ft-lbs at -40°C. For 350-360 BHN, the target impact toughness is 19 ft-lbs at -40°C. Composition G dramatically out performs other materials in the as-received condition. This composition also shows no ferrite. However, the slow aging kinetics are a potential obstacle. Composition E similarly shows better toughness, combined with slow aging kinetics. The two compositions completed to date with the fastest aging kinetics, B and D, both show poor impact toughness performance. The remaining two compositions, F and H, should intermediate behavior in both aging rate and

4. CONCLUSIONS AND FUTURE WORK

This study further emphasizes the need for continuing effort in the development of process controls for the melting of these alloys. Three major groups for aging behavior were established based on composition: fast & hard (low Mn), intermediate (high Mn & Al), and soft & slow (high Mn, low Al). These three regimes were also reflected in early impact toughness results, with fast& hard having the worst impact toughness. Future work includes completion of aging studies, the statistical analysis of data, and validation of ballistic performance. Based on this information, a large scale batch will be produced, and evaluated for weldability and machinability.

5. ACKNOWLEDGMENTS

The information, data, or work presented herein was conducted as an Advanced Vehicle Power Technology Alliance (AVPTA) "Extended Enterprise" project funded by the U.S. Army Ground Vehicle System Center (GVSC), U.S. Department of Defense (DoD), Department of the Army. AVPTA is Chartered under the auspices of the Department of Energy / DoD Memorandum of Understanding titled "Concerning Cooperation in a Strategic Partnership to Enhance Energy Security "DoD-DoE AVPTA, with additional funding from Army ManTech "Manufacturing Processes for Lightweighting Heavy Combat Vehicles". We also thank Fred Fletcher, Venkata Challa, and Laura Bartlett for their contributions to this project.

6. REFERENCES

- [1] R. J. Gerth and R. A. Howell, "What is a Ton of Weight Worth?," in *Ground Vehicle Survivability Training Symposium*, Ft. Benning, GA, 15-17 Nov., 2016.
- [2] K. Sebeck, I. Toppler, M. Rogers, K. Limmer, B. Cheeseman, R. Howell and W. Herman, "High Mn, High Al Steels for Thick Plate Armor Applications," in *GVSETS 2018*, Novi, MI, 2018.
- [3] R. Howell and J. Montgomery, *Advancements in Steel for Weight Reduction of P900 Armor Plate*, Survivability Materials Branch ARL, 2008.
- [4] O. Zambrano, "A General Perspective of Fe-Mn-Al-C Steels," Arxiv Cond-Mat 1707.01920, 2017.
- [5] I. Gutierrez-Urrutia and D. Raabe, "Influence of Al content and precipitation state on the mechanical behavior of austenitic high Mn low-density steels," *Scripta Materialia*, vol. 68, pp. 343-347, 2013.
- [6] L. Bartlett, "On the effect of silicon and phosphorous during the precipitation of K-carbide in Fe_mn-Al-C alloys," Missouri S&T Doctoral Dissertations, Rolla, MO, 2013.
- [7] I. Kalashnikov, O. Acselrad, A. Shalkevich and L. Pereira, "Chemical Composition Optimization for Austenitic Steels of the Fe-Mn-Al-C System," *J. Materials Eng. and Performance*, vol. 9, no. 6, pp. 597-602, 2000.
- [8] ASTM International, *E18 - Standard Test Method for Rockwell Hardness of Metallic Materials*, 2017.
- [9] ASTM International, *E23 - Standard Test Methods for Notched Bar Impact Testing of Metallic Materials*, 2016.
- [10] K. Limmer, D. Field, B. Cheeseman, J. Montgomery, K. Sebeck and R. Howell, "Accelerated Heat Treatment of High Mn TRIPLEX Steels for Armor Applications," in *AISTech Conference Proceedings*, Pittsburgh, PA, 2018.
- [11] R. A. Howell, *Microstructural Influence on Dynamic Properties of Age Hardenable FeMnAl Alloys*, Rolla: Missouri S&T, 2009.
- [12] O. Acselrad, I. Kalashnikov, E. Silva, M. Khadyev and R. Simao, "Diagram of Phase Transformations in the Austenite of Hardened Alloy Fe - 28% Mn - 8.5% Al - 1% C - 1.25%Si as a result of aging due to isothermal heating," *Metal Science and Heat Treatment*, vol. 48, no. 11-12, p. 543, 2006.