See discussions, stats, and author profiles for this publication at: https://www.researchgate.net/publication/349051386

# Acicular Ferrite Transformation by Isothermal Decomposition in Medium Carbon Vanadium Micro Alloyed Steel

Article · December 2019

CITATIONS O		READS 93	
3 autho	s:		
9	Abdunnaser Hamza Fadel University of Tripoli 40 PUBLICATIONS 124 CITATIONS SEE PROFILE	0	Nenad Radović University of Belgrade 77 PUBLICATIONS 546 CITATIONS SEE PROFILE
	Kamal Alhauwari University of Zawia 6 PUBLICATIONS 2 CITATIONS SEE PROFILE		

# Acicular Ferrite Transformation by Isothermal Decomposition in Medium Carbon Vanadium Micro Alloyed Steel

Abdulnaser H. Fadel<sup>1,\*</sup>, Nenad A. Radovic<sup>2</sup>, Kamal F. Alhauwari<sup>3</sup>

<sup>1</sup>Al Zawia University, Faculty of Natural Resources, Al Zawia - Libya

<sup>2</sup>Belgrade University, Faculty of Technology and Metallurgy, Kamegijeva 4, 11120 Belgrade, Serbia

<sup>3</sup> Al Zawia University, Faculty of Natural Resources, Al Zawia - Libya

\*E-mail: A. fadel@zu.edu.ly

#### Abstract

The aim of the present paper is focused on nucleation onset and development morphologies of acicular ferrite and to evaluate microstructure and mechanical properties during isothermal austenite transformation in titanium free micro-alloyed steel. Isothermal treatment was carried out in the temperature range 350 to 450 C°. These treatments were interrupted at different times between 2 and 1800 s in order to analyze the evolution of the microstructure. Yield stress was determined by compression testing on samples with final Microstructure. The metallographic evaluation was done by using optical and scanning electron microscopy (SEM) enabled determination of the nucleation onset at all treatments and subsequent on the development of acicular ferrite of isothermally treated titanium free micro-alloyed steel. The results show that during continuous cooling, dominantly acicular ferrite microstructure is formed. Main characteristics of acicular ferrite are intragranular nucleation and strongly disorganized microstructure with a larger ability to deflect cracks. Acicular ferrite is, therefore, widely recognized to be a desirable microstructure due to good mechanical properties.

**Keywords**: Micro alloyed Steel, Isothermal Transformation, Prior Austenite Grain Size, ø5x5mm Compression Test, Grain Boundary and Acicular Ferrite Nucleation.

#### **1. Introduction**

The prior austenite grain size exerts an important influence on the decomposition of austenite [1, 2]. An increase in austenite grain size leads to a reduction in the number of nucleation sites at the austenite grain boundaries indirectly favoring the intra-granular nucleation of ferrite, i.e. the formation of idiomorphic ferrite, rather than allotriomorphic ferrite. In order to obtain bainite, the austenite grain size has to be small so that nucleation from grain boundaries dominates and subsequent growth then swamps the interior of the austenite grains. By contrast acicular ferrite is nucleated intra-granularly on inclusions within large austenite grains and then radiates in many different directions. The presence of a uniform layer of allotriomorphic ferrite along the austenite grain boundaries induces the transformation of austenite in acicular ferrite instead of bainite [3-8]. The acicular ferrite is in fact intra-granularly nucleate bainite [5-7]. It is a much more disorganized microstructure with a larger ability to deflect cracks. Acicular ferrite is therefore widely recognized to be a desirable microstructure due to good mechanical properties [6]. Data related to nucleation phase of isothermal decomposition seems to be lacking; most of the published results deal with later steps (10s or longer), i.e. when the nucleation is well prolonged. Therefore, the aim of the present study is to clarify the influence of isothermal transformation temperature, time on the nucleation of ferrite and indirectly, on the development of the intragranular acicular ferrite in V micro-alloyed forging steels.

#### 2. Detailes of specimens

The chemical composition of tested steel is given in Table 1. Steel was industrially cast and hot-rolled into 19mm diameter bars. Bars were homogenized at 1250  $^{\circ}$  for 4 hours, in argon as protective

#### Al academia journal for Basic and Applied Sciences (AJBAS) volume 1/No. 2 – 2019 December

atmosphere and subsequently oil quenched. Specimens of 12 mm height were cut and austenitized at 1100 C° for 10 min in an argon atmosphere. After austenitization, specimens were isothermally held at temperatures ranging from 350 C° to 450 C° for different holding times (between 2 to 1800s) and subsequently water quenched to room temperature. The samples were prepared using standard metallographic techniques and etched in 2 % nital for their observation on optical and scanning electron microscope (SEM). After full microstructural examination, samples were cut to  $\phi$ 5x5mm dimension for compression testing. These tests were carried out on Instron servo-hydraulic tensile machine.

Element	Wt.%
С	0.256
Si	0.461
Mn	1.451
Р	0.0113
S	0.0112
Ti	0.002
V	00.99
Ν	0.0235
Fe	.bal

Table 1: The chemical composition of the material (Wt.%)

#### 3. Results

**3.1. Dissolution Temperature.** The equilibrium temperatures for complete dissolution of VN in present steel was calculated according to the equation given in Ref. [9,31], and it was estimated to be  $1100 \text{ C}^{\circ}$ .

**3.2. Prior Austenite Grain Size (PAGS).** The experimental results of the PAGS for this steel by austenitizing at 1100 C° for 10 min is 67  $\pm 3\mu$ m (Fig. 1) and is expected to enhance intra-granular AF formation rather than bainite by increasing the ratio between intra-granular and grain boundary sites [10, 11].



Fig. 1 Light optical micrographs showing revealed prior austenite grains at 1100 C°.

#### **3.3 Nucleation Onset Time**

The experimentally determined nucleation onset time is presented in table 2. As shown in figure 2, the Grain Boundary Ferrite (GBF) is the first phase to nucleate over the entire temperature range tested.

Temperature by C°	(Onset Time (s
350	7
400	2
450	2

Table 2: Experimentally determined nucleation onset time by second.



Figure. 2 SEM images and optical micrographs of Grain Boundary Ferrite GBF after 2 second at 450 C°.

#### **3.4 Acicular Ferrite Transformation**

An interlocked acicular ferrite (AF) microstructure is produced as can be seen in Fig.3. At the initial stages, the nucleation of the primary ferrite plates takes place intra-granularly at second phase particles (nonmetallic inclusions) present in the austenite, as can be seen in figure 4, which is in good agreement with [12-17,30]. The micrograph present in Figure 4 and an energy dispersive X-ray (EDX) spectrum analysis illustrate a typical active inclusion, with the corresponding chemical analysis at different points of the particle, it is identified as Ca treated manganese sulfides inclusion core covered or at least partially covered with VN or V(C, N) complex precipitate. This is in good agreement with results reported by Ochi et al and Ishikawa et al. [18, 19]. The manganese sulfides have been reported previously to be active on nucleating acicular ferrite [15, 16, 18, 20-23,30]. Moreover the inclusion size and shape is modified by Ca treatment (Morphology altered by Ca addition -in order to control and modify the shape of MnS inclusions, i.e. to get spherical shape and very low deformability). The majority of MnS inclusions in the Ca-treated steels were globular (Ca,Mn)S particles. MnS particles represented the largest contribution (60-70%) to the overall inclusion volume fraction [24]. However, the nucleation of AF plates is not restricted to inclusions, new ferrite plates can grow from pre-existing ones, as can be observed in Figure 5, which supports the view, that nucleation occurs sympathetically. In Figure 5, the sympathetic nucleation of secondary acicular ferrite plates has been observed to occur at the austenite/primary acicular ferrite interface and continue to grow within the austenite matrix until impingement occurs with other plates. The result of multiple sympathetic nucleation is an interlocking ferrite network, and after sufficient time, it results into a complex interlocking ferrite microstructure, characteristic of acicular ferrite, such as that shown in Fig. 3. The resulting microstructure with fully acicular ferrite structure in medium carbon micro-alloyed steel, with the exception of a few small bainitic zones is in good agreement with published data [23]. However, the maximum acicular ferrite

content in the present study is found for treatment carried out at 450°C. This treatment is characterized by the fully acicular ferrite formation. On the other hand, it is possible to find, in certain localized places, bainite formed at the grain boundaries, as shown in Figure 6.



Figure 3. SEM image showing the acicular ferrite interlock structure formation after 1200 s at 450 C°.



Figure 4. SEM image showing the initial stages of the primary ferrite plates takes at second phase particles and EDX spectrum of an inclusion.

# 3.5 Mechanical Testing

The compression tests were carried out at room temperature on specimens transformed isothermally at isothermal treatment was carried out at 350, 400, and 450 C° for different isothermal times. The compression equipment used in the present study is built up around a 100 KN maximum capacity of (Instron Servo-Hydraulic Testing Machine. Model 1332). The compression specimens of 5mm in diameter and 5mm in length-cylinder machined from the metallographic samples were tested in accordance with ASTM E9-89a [25,26]. These test methods cover the apparatus, specimens, and procedure for axial-load compression testing of metallic materials at room temperature. Also as

reported by [Ref. 27, 28], compression specimens with different sizes were also used. Compression tests were performed for evaluation of the offset yield strength, Rp0.2 at plastic strain of 0.2 %. The deformation speed was 0.5mm min<sup>-1</sup> corresponding to a strain rate of approximately  $4 \times 10^{-4}$  S<sup>-1</sup>. The yield strength on samples with final microstructures in this work is determined using offset method summarized in Table 2.



Figure 5. SEM image showing the sympathetic nucleation and AF interlock structure formation



Figure 6. SEM image Microstructure after completed isothermal decomposition at 450°C – bar 50µm.

Table 2. Microstructure and	vield strength ( $\sigma_{ys}$ ) after d	decomposition at different temperatu	res
ruore 2. milerostruorare una	field sheight (ovs) alter e	accomposition at aniferent temperate	100

Temperature, °C	Structure and $\sigma_{y,s}$ by Mpa
350	$\label{eq:widmanstatten} \begin{split} Widmanstatten Ferrite + Bainitic Sheaves + Sheaf Type \\ Acicular Ferrite, \sigma_{y.s=900} \end{split}$
400	$\label{eq:widmanstatten} \begin{split} Widmanstatten \ Ferrite + \ Sheaf \ Type \ Acicular \ Ferrite + \\ Bainitic \ Sheaves, \ \sigma_{y.s \ = \ 800} \end{split}$
450	Interlocking Acicular Ferrite + Sheaf Type Acicular Ferrite + WSF, $\sigma_{y.s = 680}$

#### Al academia journal for Basic and Applied Sciences (AJBAS) volume 2/No.2 – 2019 December

#### 4. **DISCUSSION**

Austenite grain size after austenitization 1100 C° /10 min is 67  $\pm$ 3µm. This austenitization is selected in order to enhance intragranular AF formation rather than bainite by increasing the ratio between intragranular and grain boundary sites [10,11] and eliminate influence of prior

austenite grain size. Final microstructures obtained after holding of 30 minutes at all three temperatures are also summarized in Table 2

At 350 C°, first phase to be nucleated are ferrite grains along prior austenite boundaries. The incubation period was 7 seconds. Due to its characteristic grain shape and low temperatures, new phase was identified as Widmanstatten ferrite (WSF). During Widmanstatten ferrite formation carbon must partition into the austenite, providing localized areas enriched with carbon. With longer times more pronounced nucleation of WSF takes place, together with some growth into austenite grains. At 10 seconds, additional grain boundaries nucleation occurs. At segments of austenite boundaries that were not covered with WSF, bainitic sheaves (BS) were nucleated. They are characterized by packets of parallel sheaves originating from brain boundaries. Bainitic sheaves are produced dominantly by shearing mechanism. Further delay up to 20 seconds leads to start of intragranular nucleation of acicular ferrite. In many cases, bainitic sheaves and acicular ferrite are differentiated only by place of origin; acicular ferrite nucleates exclusively intra granularly. Acicular ferrite formed at low temperatures follows the tendency to form sheaves composed of parallel plates. This behavior is introduced by differences in the carbon concentration profiles in the parent austenite in the front of the interface of the Widmanstatten ferrite grains. As it is suggested [21,29], at initial stages for the transformation at 350°C, the carbon concentration in austenite close to the tips of primary plates is lower than at the faces (side direction).

Therefore, instead of growth in side direction, nucleation on the tips of present sheaf is favoured, due to carbon enrichment on side direction. Therefore, final microstructure consists of Widmanstatten Ferrite + Bainitic Sheaves + Sheaf Type Acicular Ferrite (WSF+BS+STAF).

At 400°C, austenite decomposition follows, generally, the same pattern as at 350°C. Incubation period is shorter, i.e. Widmanstatten ferrite nucleates after 2 seconds. Bainitic sheaves are observed after 10 seconds, while acicular ferrite is observed after 15seconds. The main difference is that bainitic sheaves are observed sporadically, while sheaf type acicular ferrite is dominant microconstituent. Therefore, final microstructure consists of Widmanstatten Ferrite + Sheaf Type Acicular (+Ferrite Bainitic Sheaves) - WSF+STAF (+BS).

At 450°C, small portion of grain boundary ferrite is evaluated after 2 seconds incubation time. On the other hand, after 10 seconds, very pronounced nucleation of acicular ferrite starts. Due to increase in temperature, together with sheaf type acicular ferrite, interlocking acicular ferrite is also intragranularly nucleated.

The yield strength is the most important value for structure design because it determines the stress at which the material begins to deform plastically after dislocations start the glide. Highest value of 900 MPa is determined for samples transformed at 350°C. With increase of decomposition temperature, yield stress decreases to 800 and 680 MPa, for 400 and 450°C, respectively. Due to dominantly displacive nature of isothermal decomposition at lower temperatures, this behavior can be related to dislocation strengthening associated with the presence of hard phases such as bainite. As have been reported previously, the dislocation density of the ferrite increases with decreasing transformation temperature. This observation can be associated with a high strain-hardening rate imposed by bainite.

# 5. CONCLUSION

Results indicate presence of three dominant morphologies, Widmanstatten ferrite (WSF), bainitic sheaves (BS) and sheaf type acicular ferrite (STAF). WSF was first nucleated at grain boundaries, at all temperatures. In some cases, bainitic sheaves are also present at prior austenite grain boundaries. This feature decreases with increase of temperature. As time elapsed, intragranular nucleation of acicular ferrite started. Acicular ferrite has typical sheaf type morphology, differing from bainitic sheaves only

in nucleation place. Yield strength decreases from 900 MPa to  $\sim$  700 MPa with increase of holding temperature from 350°C to 450°C. This behavior is attributed to decrease in dislocation density with increase of temperature in the case of displacive transformation.

### References

[1] M.A. Linaza, J.L. Romero, J.M. Rodriguez-Ibabe, J.J.Urcola, Scripta Met. Mater., 32 (3) (1995) 395–400.

- [2] A. Echeverria, J.M. Rodriguez-Ibabe, Scripta Mater.,41 (2) (1999) 131–136.
- [3] A. Fadel, N. Radović, Int. J. of Eng. And Information Technol. (IJEIT), 2 (2015) 37 39.
- [4] M.A. Linaza, J.L. Romero, J.M. Rodriguez-Ibabe, J.J.Urcola: Scripta Met. Mater. 29 (9) (1993) 1217–1222.
- [5] I. Madariaga, I. Gutierrez, Materials Science Forum., 284-286 (1998) 419-426.
- [6] A. Fadel, N. Radović, Int. J. of Eng. And Information Technol. (IJEIT), 3 (2017) 170 178.
- [7] A. Echeverria, J.M. Rodriguez-Ibabe, Scripta Mater., 41 (2) (1999) 131–136.
- [8] M.J. Balart, C.L. Davis, M. Strangwood, Mater. Sci. Eng., A284(2000) 1–13.
- [9] A. Fadel, D. Glišić, N. Radović, Dj. Drobnjak, J. Mater. Sci. Technol., 28 (2012),1053-1058.
- [10] C. Capdevila, F. G. Caballero. C. Gracia-Mateo and C.Garcia de Andres: Mater. Trans., 45 (2004), 2678.
- [11] C. Capdevila, F. G. Caballero, and C. Garcia de Andres: Mater. Sci. Technol., 19 (2003) 195-201.
- [12] S.S. Babu and H.K. D. H. Bhadeshia: Mat. Trans., JIM 32 (8) (1991) 679-688.
- [13] H.K.D.H. Bahdeshia: Bainite in Steels, The Institute of Materials, London, 2001, p.237.
- [14] A. Khodobandeh, M. Jahazi, S. Yue and P. Bocher: ISIJ Int., 45 (2) (2005) 272-280.
- [15] T. Furuhara, T. Shinyoshi, G. Miyamoto, J. Yamaguchl, N. Sugita, N. Kimura, N. Takemura and T. Maki: ISIJ Int., 43 (12) (2003) 2028-2037.
- [16] C. Capdevila, F.G. Caballero, and C. Garcia de Andres: Met. and Mat. Trans.A., 32A (2001) 1591-1597.
- [17] M. Diaz-Fuentes, I. Gutiérrez: Mat. Sci and Eng., A363(2003) 316-324.
- [18] T. Ochi, T. Takahashi and H. Takada: Mechanical working and Steel Processing Conf. Proe., ISS-AIME, Warrendale, PA, USA, 1988, p. 65-72.
- [19] F. Ishikawa, T. Takahashi, T. Ochi: Metall. Mater. Trans., 25A (5) (1994) 929-936.
- [20] S. Zajac: Mat. Sci. Forum., 500-501(2005) 75-86.
- [21] I. Madariaga, I. Gutierrez, H.K.D.H. Bhadeshia:Metall. Trans., A 32A (9) (2001) 2187-2197.
- [22] I. Madariaga and I. Gutierrez: Acta mater., 47 (3) (1999) 951-960.
- [23] I. Madariaga, J. L. Romero, and I.Gutierrez: Met. And Mat. Tran., 29 A (1998) 1003-1015.
- [24] M.J. Balart, C.L. Davis, M. Strangwood: Mat. Sci. AndEng., A328 (2002)48-57.
- [25] F. G. Caballero, H.K.D.H. Bhadeshia, K. J. A. Mawella, D. G. Jones, P. Brown. Mate. Sci. and Tech 18 (2002) 279.
- [26] S. Zajac, T. Siwecki, B. Hutchinson, R. Lagneborg: ISIJ Int.38 (1998) 1130-1139.
- [27] F. Ishikawa, T. Takahashi ISIJ Int. 35(1995)1128-1133.
- [28] T. Siwecki, J. Eliasson, R. Lagneborg, B. Hutchinson. ISIJ. 50 (2010) 760-767.
- [29] I. Madariaga, I. Gutierrez, C. G. Deandres and C. Capdevila, Scripta Mater., 41 (1999) 229.
- [30] A. Fadel, D. Glišić, N. Radović, Dj. Drobnjak, J. Min. Metall. Sect. B-Metall., 49(3) B (2013) 237.
- [31] D. Glišić, N. Radović, A. Koprivica, A. Fadel, Dj. Drobnjak, ISIJ International 50 (4) (2010) 601.