Effect of Isothermal Heat Treatment Temperature and Time on Microstructure and Mechanical Properties of Titanium Free Medium Carbon Micro Alloyed Steel

Abdulnaser H. Fadel[1*], Nenad A. Radovic[2], Dragomir Glišić[2]

[1]Al Zawia University, Faculty of Natural Resources, Al Zawia - Libya

[2] Belgrade University, Faculty of Technology and Metallurgy

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

Abstract

The main goal of the current paper is focused to investigate the effect of isothermal heat treatment temperature and time on microstructure and strength in a medium carbon vanadium titanium free micro alloyed steel. Isothermal heat treatment was carried out in the temperature range 350 to 600 C° at different holding times varying from 2s to 1200s followed by water quenching. Samples were investigated using optical microscope (OM) and scanning electron microscopy (SEM) paired with energy dispersive spectroscopy (EDS) and by compressive testing using a servo-hydraulic testing machine. The results show that, the final microstructure of samples held at high temperatures (550 and 600°C) consists of polygonal intra-granularly nucleated ferrite idiomorphs, combined with grain boundary ferrite and pearlite were produced and followed by retained austenite that transformed to martensite upon quenching (incomplete transformation phenomenon). At intermediate temperatures (450 and 500 °C) an interlocked acicular ferrite (AF) microstructure is produced, hence acicular ferrite becomes prevalent in the microstructure at (450 °C). The microstructure after the heat treatment at 500°C consists coarse nonpolygonal ferrite grains separated by pearlite colonies. However, at low temperatures (400 and 350°C), the final microstructure of the samples held at 350°C consists of bainitic sheaves, where the sheave of parallel acicular ferrite plates, similar to bainitic sheaves but intra-granularly nucleated were observed, which called in some references as sheaf type acicular ferrite for samples isothermally treated at 400°C. Yield stress was determined by compression testing on samples with final Microstructure, the results show that, the observed change in the microstructure is related by a marked decrease of compressive yield strength, approximately from 1000 to 700 MPa.

Keywords: Medium Carbon Microalloyed Steel, Isothermal Transformation, Grain Boundary Ferrite, Bainite Sheaves, Acicular Ferrite, Pearlite, ø5x5mm Compression Test.

1.Introduction

The microstructure and properties are of significant importance in many areas, for example, in the development of new alloys, in structure property correlation, in steel quality evaluation, and in failure analysis. So, the structural changes play an important role in determining the final microstructure and properties of high strength low alloy steels. Depending on the alloying content and the cooling rate, microstructure of continuously cooled medium carbon micro alloyed steel from the temperature of hot working could be in general ferritic-pearlitic or bainitic, but may contain acicular ferrite, Widmanstätten ferrite, different morphologies of grain boundary and intragranular ferrites, retained austenite and martensite [1,2]. Acicular ferrite forms by the same reaction as bainite, except that the nucleation site shifts from the grain boundaries to the grain interiors, at the second phase particles [1,3-5]. Although satisfactory strength levels alongside with the fatigue resistance has been attained, toughness of the ferritic-pearlitic medium carbon micro alloyed steels is still considerably lower comparing to that of quenched and tempered steels. Results of previous investigations [5-8] indicate that fine interlocked structure of acicular ferrite have beneficial effect on toughness of the medium carbon micro alloyed steels. Also, several studies [9-12] have shown that such microstructures are better suited to deflect propagation of cleavage cracks and therefore more desirable from toughness point of view in comparison to bainitic sheaves. Vanadium-nitride particles, beside their role in precipitation

hardening, are found to be highly effective as a preferential place for intragranular nucleation of acicular ferrite [13-15]. This way, vanadium addition together with high nitrogen levels in the steel favors the formation of acicular ferrite at the expense of bainite [15], and thus acts in direction of fracture toughness improvement. However, there is a question of strength of the steels with bainitic or acicular ferrite structure, which seems to be relatively low compared to the medium carbon micro alloyed steels with ferrite-pearlite structure [4]. There is therefore an interest to evaluate the contribution of individual microstructural components to the overall strength of the medium carbon micro alloyed steels, in order to find the way to improve their mechanical properties and to further expand their application. In order to estimate the contribution of the individual microconstituents to the overall strength of the steel, investigation of transformational behavior of the steel on isothermal heat treatment seems to be a convenient method. A range of microconstituents attained at different temperatures of isothermal holding in this current paper were used with the aim to evaluate their impact on the strength of a medium carbon vanadium titanium free micro alloyed forging steel with high level of nitrogen content.

2.Experimental Procedures

2.1 Materials

Commercial vanadium medium carbon micro alloyed forging steel without titanium addition have been studied. The chemical compositions of these steel are given in Table 1. The steel was industrially casted by full-scale casting followed by hot forging and hot rolling into 19mm diameter bars. In order to break the dendritic structure, bars were homogenized in laboratory furnace at 1250 °C for 4 hours, in argon as protective atmosphere and subsequently oil quenched. Specimens of 12mm height were cut and austenitized at 1100 °C for 10 min in an argon atmosphere. After, austenitization, specimens were isothermally held in salt bath for further isothermal treatment at temperatures ranging from 350 C° to 600 C° for different holding times varying from 2 to 1200 second followed by water quenching.

Table 1: Chemical composition of the experimental steel (wt.%)

C	Si	Mn	P	S	Cr	Ni	Cu	Al	Mo	Ti	V	Nb	N
0.256	0.416	1.451	0.0113	0.0112	0.021	0.149	0.183	.038	0.023	0.002	0.099	0.002	0.0235

2.2 Microstructural Characterization

General characteristics of microstructure were observed under optical microscope from samples isothermally treated and polished following standard procedure and etched by 2% NITAL. In order to reveal the fine features in detail, etched samples were also observed by a Field Emission Gun Scanning Microscope (FEGSEM). Elemental analysis in a fine scale was carried out with an Electron Probe Micro-Analysis (EPMA). Camera SX 100 instruments were used for such purpose with an acceleration voltage of 15 kV, beam current 20 mA and with beam diameter of 1 µm.

2.3 Mechanical Testing

The compression tests were carried out at room temperature on specimens transformed isothermally from 350 C° to 600 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 [16,17]. These test methods cover the apparatus, specimens, and procedure for axial-load compression testing of metallic materials at room temperature. Also as reported by [18,19], compression specimens with different sizes were also used. Compression tests were performed for evaluation of the offset yield strength, $Rp_{0.2}$ at plastic strain of 0.2 %. The deformation speed was 0.5mm min^{-1} corresponding to a strain rate of approximately 4×10^{-4} S^{-1}.

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. [20], and it was estimated to be 1100 C°.

3.2 Prior Austenite Grain Size (PAGS)

Because the austenite grain size control is important factor in development of the final mechanical properties of the product. The experimental results of the PAGS for this steel by austenitizing at 1100 C° for 10 min is 60 ±3μm, and is expected to enhance intra-granular AF formation rather than bainite by increasing the ratio between intra-granular and grain boundary sites [21-23].

3.3 Nucleation Onset Time

The incubation time is the minimum time at which it is possible to find some ferrite nucleated at the austenite grain boundary. Therefore, as represented in table 2, the temperature at which the incubation time for ferrite nucleation is at minimum value is approximately the same (2s) for both temperatures (400 and 450 °C). The experimentally determined nucleation onset time for all isothermal treatment is presented in table 2. The Grain Boundary Ferrite (GBF) the first phase to nucleate over the entire temperature range tested, is shown in Figure 1 (d) and is represented by two types of transformation describing the effect of temperature on nature of transformation. Second phase is related to intra-granularly nucleated ferrite (IGF) and the third to the pearlite (P) phase as shown in figure 1. The GBF and IGF phases are divided into the high temperature and the low temperature segments as consequence of either displacive or diffusion nature of transformation.

Table 2. Experimentally determined nucleation onset time by second for different phases observed.

Temperature by ° C	Phases observed				R_f
	GBF	IGF	BS	P	
350	7	Nil	10	Nil	600
400	2	Nil	20	Nil	600
450	2	Nil	Nil	Nil	1200
500	3	10	Nil	80	> 1200
550	5	20	Nil	45	> 1200
600	7	30	Nil	30	> 1200

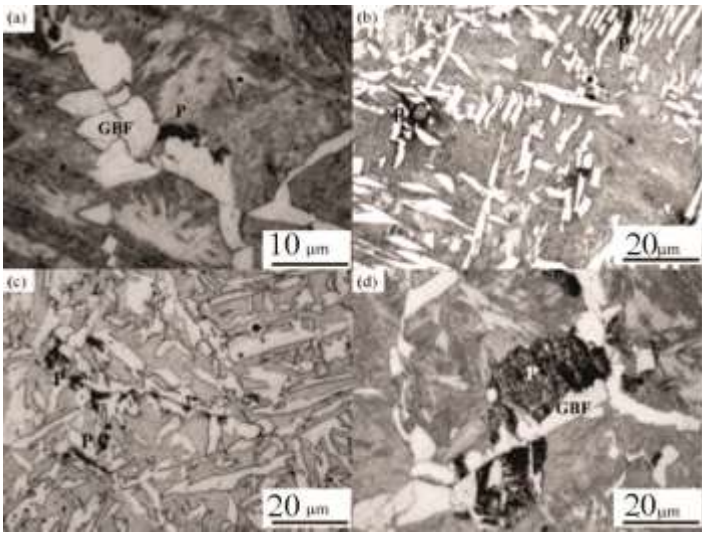

Figure 1. Optical micrographs of V-N-Ti free steel showing the onset of pearlite: (a) 30s at 600 °C.

(b) 45s at 550 °C. (c) 80s at 500 °C (d) 60s at 600 °C.

3.4 Incomplete Transformation

As represented in table 2. The transformation after1200s of isothermal treatment at 550 and 600 ˚C reveals that a fraction of austenite remains untransformed (UA) i.e (Reaction finish time R_f > 1200 second), as can be seen in Fig.3(a) This phenomenon has been described by Bhadeshia and another studies [8,12, 23 – 26, 29-31] and is known as incomplete reaction phenomenon.

3.5 Strength

The yield strength on samples with final microstructures in this work is determined using offset method summarized in Table 3. Compressive yield strength dependence on temperature of isothermal heat treatment as represented in figure 2 by (closed line curve). The compressive yield stress ($\sigma_{Y.S}$) decreases with temperature starting from 350 °C and reaches the minimum at 450 °C and 500 °C further, at 550 °C and 600 °C, values are increased.

Table 3. Experimentally determined Compressive yield Stress At different isothermal transformation temperatures

Temperature, °C	Compressive Yield Strength, MPa
350	900
400	800
450	680
500	680
550	700
600	700

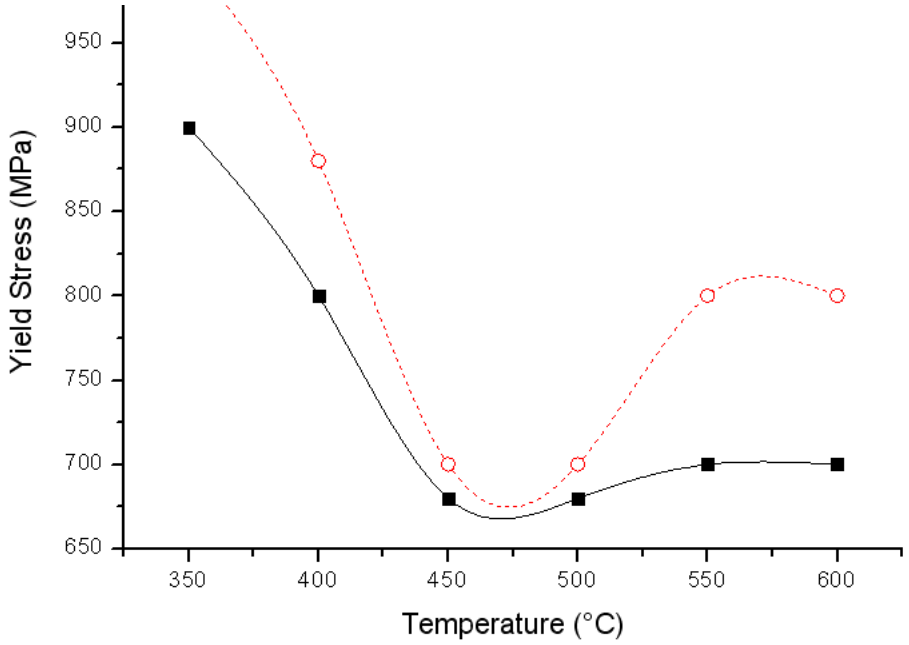

Fig 2. Compressive Yield stresses (σ_{02}) on different transformation temperatures for V-N-Ti free micro alloyed steel (closed line curve symbol).

3.5 **Microstructure**

The microstructures obtained after 1200s of isothermal treatments at 350, 400, 450, 500, 550 and 600 ℃ are presented in figure 3 and figure 4 respectively describing the effects of diffusional and displacive nature of transformation.

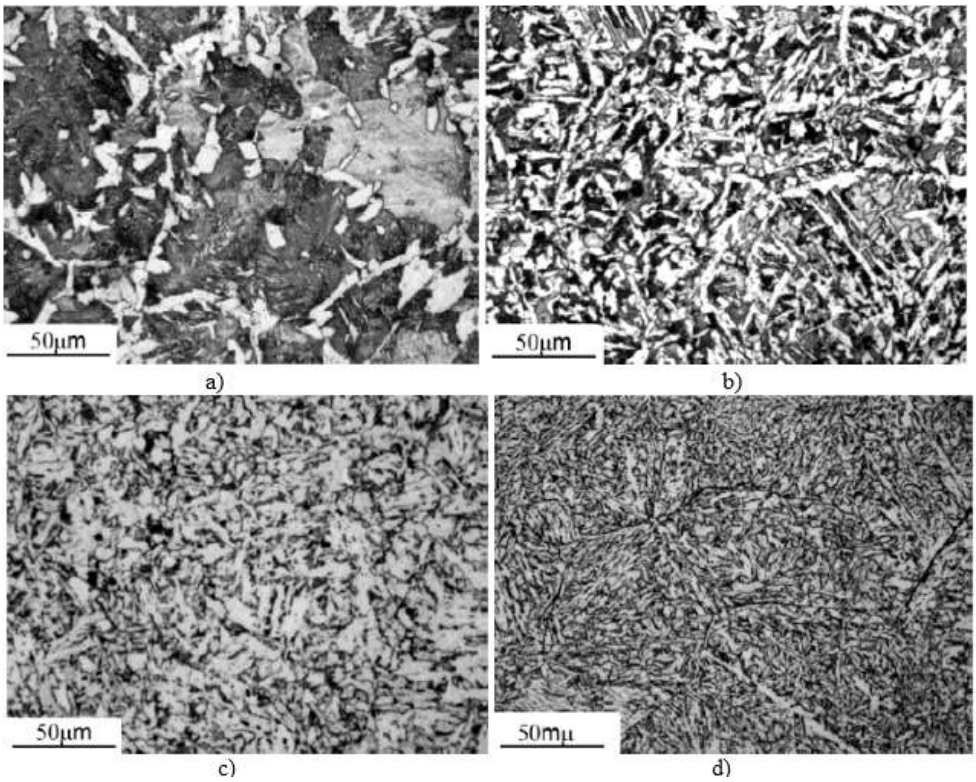

Fig. 3. Optical micrographs showing the microstructures after 1200s of isothermal transformation temperatures. Thus, for (a) 600 °C (b) 550 °C (c) 500 °C (d) 450 °C.

4 Discussion

4.1 Microstructure

Three different morphologies have been observed depending upon the isothermal treatment temperature. At high temperature the austenite transforms entirely by diffusional or reconstructive mechanism (Allotriomorphic Ferrite, Idiomorphic Ferrite, Massive Ferrite or Pearlite). However, at low temperature it transforms by displacive mechanism (Widmanstatten Ferrite WSF, Bainite, Acicular Ferrite or Martensite). Firstly, at high temperatures, (≥ 550 °C) intra-granularly nucleated ferrite combined with grain boundary ferrite (GBF) and pearlite (P) are produced, as shown in figures 1. The intragranular ferrite is characterized by polygonal idiomorphic (IGF) Ferrite. The idiomorphic ferrite nucleates intra-granularly at the inclusions distributed inside the austenite grains. The transformation after 1200s of isothermal treatment at 550 and 600 °C reveals a fraction of untransformed austenite, Fig.3(a, b). Second type of intragranular ferrite morphologies occurs at intermediate temperature (450 and 500 °C). The final microstructure of the samples isothermally heat treated at 500°C (Fig. 3c) consists of non-polygonal, coarse and somewhat elongated ferrite grains, separated by pearlite nodules. However, an interlocked acicular ferrite (AF) microstructure is produced as can be seen in Fig. 5(a-c), Fig 3(d). However, the temperature at which the incubation time for ferrite nucleation is at minimum is approximately at 450 °C (Table 2). The incubation time is the minimum time at which it is possible to find some ferrite nucleated at the austenite grain boundary. The, maximum acicular ferrite content in the present steel 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 5(d). The onset of pearlite at different a treatment temperature (≥ 500 °C) is illustrated in Figure 1. The third type of intragranular ferrite morphology exist as temperature is decreased to 400 and 350 °C, mainly two morphologies are observed bainite sheaves (BS) and sheaf type acicular ferrite (STAF) with (WSF) in some localized places as demonstrated in Fig. 4 and Fig. 6. The acicular ferrite sheaf morphology is frequently observed when the isothermal transformation time is increased at 400 °C as illustrated in Fig. 6(c, d) and 4 (b). Two different morphologies are present at the beginning of the transformation, Bainite sheaves BS and sheaf type acicular ferrite STAF. The origin of Bainitic sheaves are exclusively the grain boundaries. When, the isothermal treatment times is increased a new intragranular morphology known as the sheaf type acicular ferrite (STAF) [7] is observed as can be seen in Fig. 6.

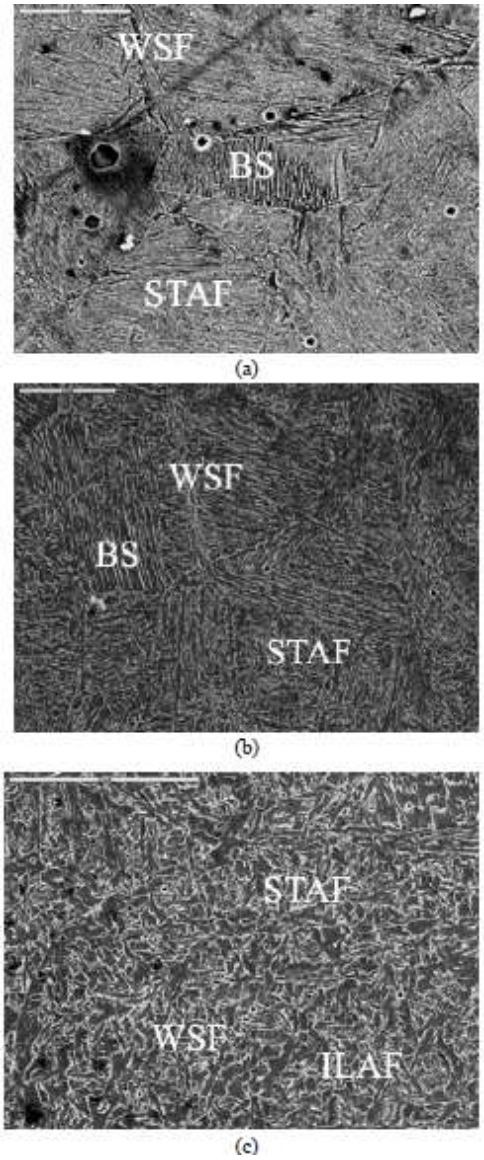

Figure 4. SEM microphotographs showing the microstructures after 1200s of isothermal transformation temperatures. Thus, for (a) 350 °C – bar 20μm; (b) 400 °C – bar 20μm; (c) 450 °C – bar 50μm.

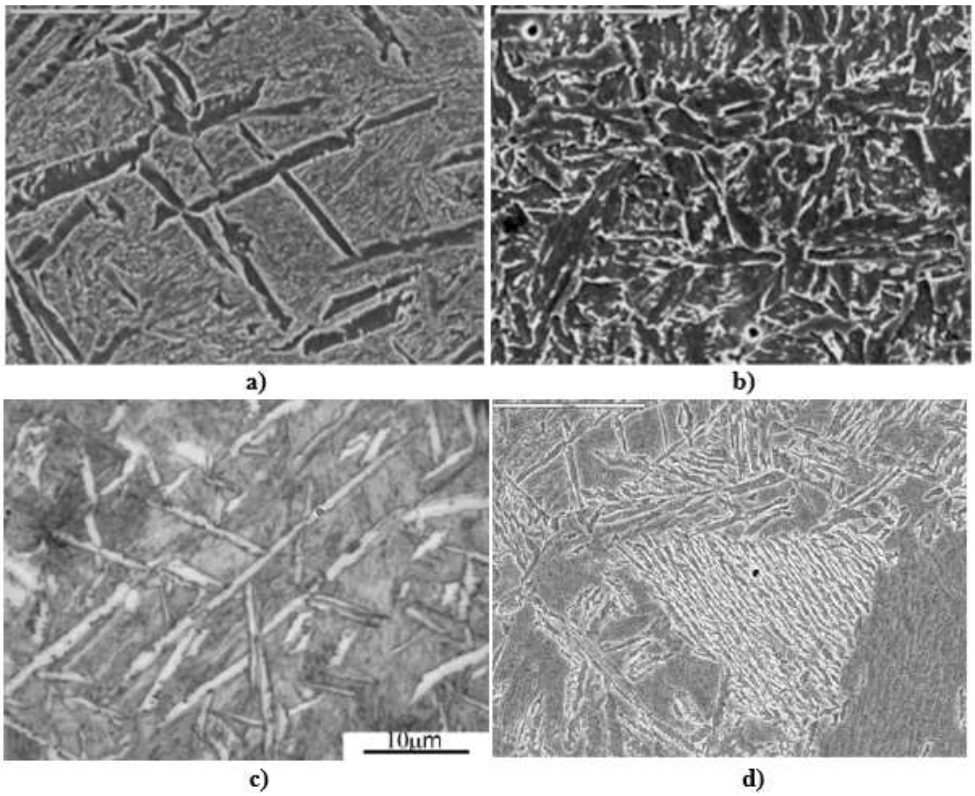

Figure 5. SEM and O.M images (a, c) Showing the sympathetic nucleation of AF after 20s of isothermal treatment at 450°C (b) SEM image showing AF interlock structure formation after 1200s of isothermal treatment at 450°C. (d) SEM image showing localized places of bainite.

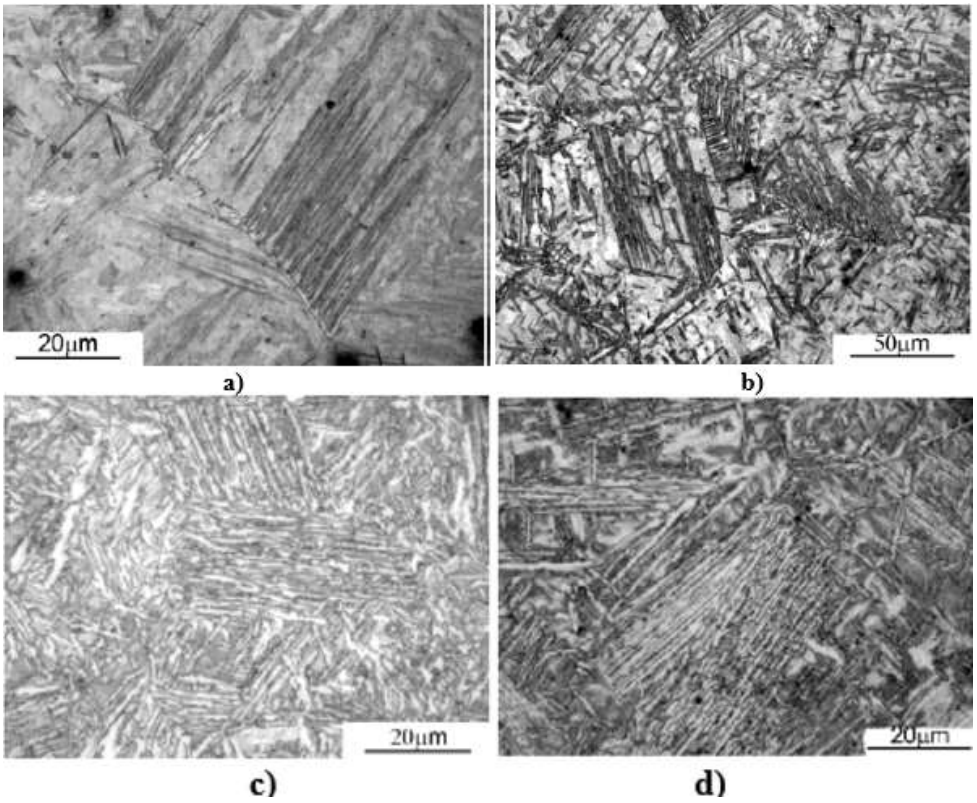

Figure 6. (a - d) Optical Micrographs (OM) Showing (a) BS onset after 10s at 350°C.(b) BS after 20s at 350°C. (c) STAF exist after 30s at 400°C (d) STAF exist after 20s at 400°C.

4.2 Yield Strength

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. Compressive yield strength dependence on temperature of isothermal heat treatment Fig.2. (closed line curve symbol). 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, 450 and 500 °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. Low strength of specimen subjected to isothermal decomposition at 500°C could be associated with higher amounts of coarse ferrites with low dislocation density. Despite coarser microstructure and assumed lower dislocation density compressive YS remains at the same level as for 450°C, probably due to the presence of fine pearlite. Increase of compressive YS for the samples held at 550°C and 600°C could be related to the increased content of fine pearlite in the microstructure [27]. However, detected increase of the strength was also affected by the presence of the martensite in the structure that formed upon quenching from the temperature of isothermal heat treatment (light gray area at the micrograph in Figure 3a and 3b). This suggests that finish of transformation was delayed to longer times, which could be explained by increased hardenability of the steel, as a result of relatively high content of carbon and the additional influence of substitutional alloying elements. The enrichment of austenite on carbon during transformation inhibits further progress of diffusional decomposition of austenite to pearlite [8,23,26]. Among other substitutional elements, such as Cr or Ni, it is known that V in austenite solid solution markedly increases hardenability of steels [28]. Having in mind relatively high content of V in the steel and its higher solubility in austenite than in ferrite [28], a quantity of free excess V in solid solution could be expected, which could explain observed retardation of austenite decomposition and the delayed finish of transformation at 550°C and 600°C.

5. Conclusions

The aim of this work was to evaluate microstructure and mechanical properties after isothermal decomposition of austenite in a medium carbon vanadium titanium free micro alloyed steel. The tested steel contained 0.256%C, 0.12%V and 0.229%N. Samples were reheated at 1100 °C for 10 minutes and then isothermally treated in salt bath at 350, 400,450,500,550 and 600 °C. Microstructure was revealed using light and SEM microscopy. Yield stress was determined by compression testing on samples with final microstructure. At 350 to 400 °C the 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 800 MPa with increase of holding temperature from 350 to 400 °C. This behavior is attributed to decrease in dislocation density with increase of temperature in the case of displacive transformation. Formation of acicular ferrite structure at 450°C and 500°C is accompanied by a noticeable decrease of compressive YS to 680 MPa. However, the decomposition of the austenite to ferrite and pearlite was observed at 550-600°C is characterized by the delay of transformation finish, due to the influence of relatively high content of C and substitutional alloying elements, Cr and Ni, but in particular V on retardation of diffusion in austenite.

References

[1] A. Fadel**,** Al academia journal for Basic and Applied Sciences (AJBAS) 1 (2) Dec. (2019) 1- 7.
[2] M. Gomez, L. Rancel, E. Escudero, S.F. Medina, J. Mater. Sci. Technol. 30 (2014) 511–516.
[3] C. Garcia de Andres, C. Capdevila, F.G. Caballero, D. San Martin, J. Mater. Sci. 36 (2001) 565–571.
[4] A.R. Khodabandeh, M. Jahazi, S. Yue, P. Bocher, ISIJ Int. 45 (2005) 272–280.
[5] S.S.Babu, H.K.D.H. Bhadeshia, JIM 32 (1991) 679–688.
[6] D. Glišić, N. Radović, A. Koprivica, A. Fadel, D. Drobnjak, ISIJ Int. 50 (2010) 601–606.
[7] I. Madariaga, I. Gutierrez, H.K.D.H. Bhadeshia, Metall. Mater. Trans. A 32 (2001) 2187–2197.
[8] H.K.D.H. Bhadeshia, Bainite in Steels, IOM Communications Ltd, London, 2001.
[9] C. Capdevila, J. P. Ferrer, C. García-Mateo, F. G. Caballero, V. López, C. García de Andrés, ISIJ Int. 46 (2006)1093–1100.
[10] J. Fernández, S. Illescas, J.M. Guilemany, Mater. Lett. 61 (2007) 2389–2392.
[11] J. Shim, Y. Oh, J. Suh, Y.W. Cho, J. Shim, J. Byun, Acta Mater. 49 (2001) 2115–2122.
[12] H.K. D. H. Bhadeshia, R.W.K. Honeycombe, Steels: Microstructure and Properties, Elsevier Ltd, London, (2006).
[13] F. Ishikawa, T. Takahashi, T. Ochi, Metall. Mater. Trans. A 25 (1994) 929–936.
[14] J. Hu, L.X. Du, H. Xie, X.H. Gao, R.D.K. Misra, Mater. Sci. Eng. A 607 (2014) 122–131.
[15] C. Garcia-Mateo, J. Cornide, C. Capdevila, F.G. Caballero, C. Garcia de Andres, ISIJ Int. 48 (2008) 1276–1279.
[16] F. G. Caballero, H.K.D.H. Bhadeshia, K. J. A. Mawella, D. G. Jones, P. Brown. Mate. Sci. and Tech 18 (2002) 279.
[17] S. Zajac, T. Siwecki, B. Hutchinson, R. Lagneborg: ISIJ Int.38 (1998) 1130-1139.
[18] F. Ishikawa, T. Takahashi ISIJ Int. 35(1995)1128-1133.
[19] T. Siwecki, J. Eliasson, R. Lagneborg, B. Hutchinson. ISIJ. 50 (2010) 760-767.
[20] H. Adrian: Proc. of Int. Conf. Microalloying ’95, ISS, Warrendale, PA, USA,1995, p. 285.
[21] C. Capdevila, F. G. Caballero. C. Gracia-Mateo and C. Garcia de Andres: Mater. Trans., 45 (2004), 2678.
[22] C. Capdevila, F. G. Caballero, and C. Garcia de Andres:Mater. Sci. Technol., 19 (2003) 195-201.
[23] H. Bhadeshia, D. Edmonds, bainite transformation in Si steel, Metall. Trans. A10 (1979) 895–907.
[24] T. Ochi, T. Takahashi: “Improvement of the Toughness of Hot Forged Products “Through Intra-granular Ferrite Formation”, in 30th Mechanical Working and Steel Processing Conference Proceedings, Vol. XXVI, ISS-AIME, Warrendale, Pennsylvania, (1988) 65-72.
[25] W. Roberts, A. Sandberg,T. Siwecki, Precipitation of V(C,N) in HSLA Steels Micro alloyed with V, Proc. Conf. Vanadium Steels, Krakow, Vanitec (1980) D1-D12.
[26] M. Diaz-Fuentes, I. Gutierrez, Mat. Sci. and Eng. A363 (2003) 316-324.
[27] E. Pereloma, D. V Edmonds, Phase Transformations in Steels, 2012.
[28] L. Rune, H. Bevis, S. Tadeusz, S. Zajac, Artic. Scand. J. Metall. (1999) 102.
[29] A. Fadel, D. Glišić, N. Radović, D. Drobnjak, J. Min. Metall. Sect. B-Metall. 49 (3) B (2013) 237.
[30] A. Fadel**,** Al academia journal for Basic and Applied Sciences (AJBAS) 2 (2) Dec. (2020) 1- 6.
[31] A. Fadel**,** N. Radović, Int. J. of Eng. And Information Tech. (IJEIT),3 (2), (2017) 170-178.