SINTERED METALS AND ALLOYS

THE EFFECT OF VANADIUM AND TITANIUM
ON MECHANICAL PROPERTIES OF MICROALLOYED PM STEEL

S. Gunduz,[1,5] M. A. Erden,[2] H. Karabulut,[3] and M. Turkmen[4]

The effect of Ti and V additives on the microstructure and mechanical properties of microalloyed powder metallurgy (PM) steels is investigated. The microstructure of microalloyed PM steels is characterised with the help of optic microscope, SEM and EDS. The results showed that the addition of Ti and V elements has a beneficial effect on the improved mechanical properties. Ti–V microalloyed PM steels can be used to take advantage of improved grain refining propensity of titanium, whilst allowing vanadium to be used as dispersion strengtheners and to enhance the hardenability and transformation characteristics.

***Keywords**: powder metallurgy, microallyed steels, microstructure.*

INTRODUCTION

Microalloyed steels utilize the chemical composition design of low carbon content and are microalloyed with V, Nb, and Ti, and other additives, such as Mo, B, etc. The increase of strength is attributed to grain refinement, solid-solution strengthening, dislocation strengthening and precipitation hardening. Moreover, the precipitation hardening attracts more and more attention of researchers [1]. Micro additives of V, Nb, Ti, and their combination improve the strength by formation of fine precipitates (precipitation strengthening) and/or by being dissolved in the ferrite matrix (solid solution strengthening). Also, they induce grain refinement through suppression of austenite grain boundary migration through solute drag effect and precipitation pinning effect [2–6], such that fine ferrite grains will be produced eventually upon transformation.

In modern microalloyed steels the requirements for specific properties may call for the use of more than one microalloying element. Ti–V steels, where titanium is used for grain refinement and vanadium for dispersion strengthening, can serve an example. Ti–V steels have commonly been used with a dual role in controlling the hot-rolled microstructure (due to the marked retardation of austenite recrystallization) and as a dispersion strengthener, the vanadium in this case being used to augment the dispersion strengthening [7, 8].

[1]Karabük University, Technnology Faculty, Department of Manufacturing Engineering, 78050, Karabük, Turkey. [2]Karabük University, Institute of Science and Technology, Department of Manufacturing Engineering, Karabük, Turkey. [3]Karabük University, Karabük Vocational School, 78050, Karabük, Turkey. [4]Kocaeli University, Hereke Vocational School, Department of Metallurgy, Kocaeli-Hereke, Turkey.

[5]To whom correspondence should be addressed; e-mail: sgunduz@karabuk.edu.tr.

More recently, Ti–V and Ti–Nb steels are being used to take advantage of improved grain refining propensity of titanium, whilst allowing vanadium and niobium to be used as dispersion strengtheners and to enhance hardenability and transformation characteristics. The behavior of microalloying elements can be modified by the presence of another such element, and the changes are dependent on the particular elements considered, but in principle can be divided depending on whether the two nitrides (or carbides) show mutual insolubility, or whether they show mutual extended solubility [7, 9].

Pandit et al. [10] investigated the strain-induced precipitation kinetics of carbonitrides in both 0.035 w/o Nb–0.125 w/o V and 0.036 w/o Ti–0.13 w/o V microalloyed steels based on a stress relaxation phenomenon. They found that the precipitates were (Nb,V)(C,N) and (Ti,V)(C,N) in Nb–V and Ti–V steels, respectively, due to the relatively large amount of V concentration in the two steels. They also reported that the precipitation kinetics of (Nb,V)(C,N) in Nb–V steel were faster than those of (Ti,V)(C,N) in Ti–V steel because of the low solubility of Ti in γ and the reduced amount of solute Ti atoms in γ caused by undissolved TiN particles during solution treatment in the Ti–V steel.

Powder metallurgy (PM) provides great advantages for manufacturing large series of small size and relatively complex shape components, and therefore largely increases the utilization ratio of materials and reduces machining cost. The homogeneous and fine microstructure can also be obtained in this way [11–13].

The purpose of this study is to examine the effect of V and Ti on the microstructure and mechanical properties of sintered PM steels. For this purpose, V and Ti were added to several microalloyed PM steels. The microstructure was characterized and mechanical properties were measured in the sintered condition to assess the role of precipitation strengthening and grain refinement.

MATERIALS AND EXPERIMENTAL PROCEDURE

Fe, V, and Ti powders of ≤74, 44, and 149 μm in size (Aldrich supply) were used for the experiment. An analysis indicated that the purity of Fe, V, and Ti was 99.9%, 99.5%, and 99.7%, respectively. Figure 1 shows electron micrographs of the powders. The required mass of Fe–0.25C (Alloy 1), Fe–0.25C–0.05V–0.05Ti (Alloy 2), Fe–0.25C–0.075V–0.075Ti (Alloy 3), and Fe–0.25C–0.1V–0.1Ti (Alloy 4) powders was accurately weighed and
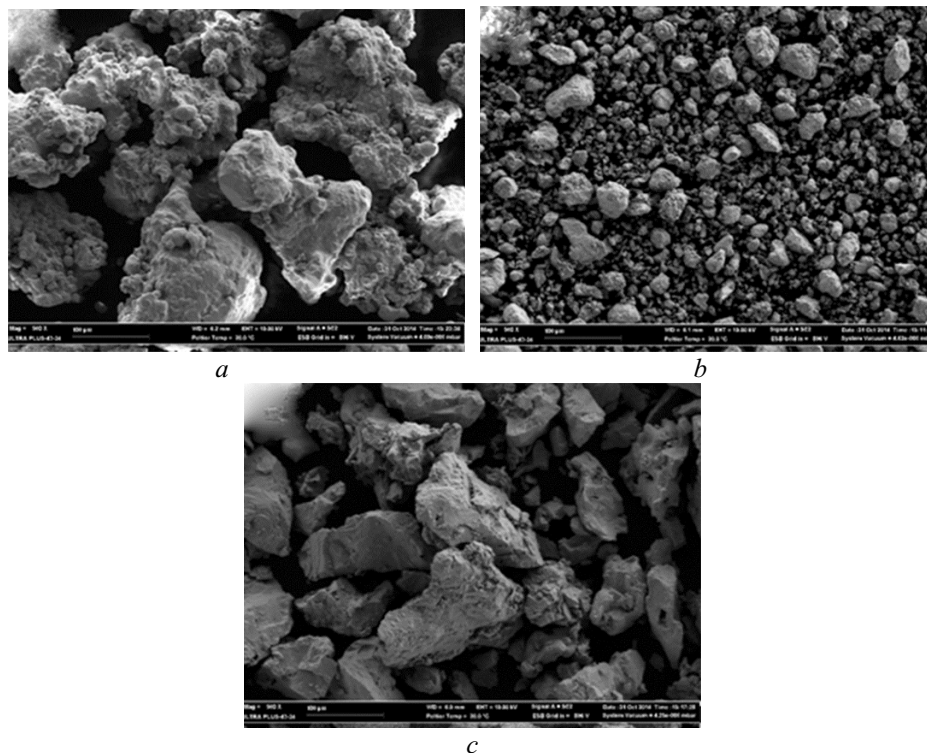

a

b

c

Fig. 1. SEM micrographs of Fe 75 μm (*a*), V 44 μm (*b*), and Ti 149 μm (*c*) powders

mixed in an industrial conical mixer for 1 h. Graphite additives were 0.45% to reach carbon concentration of 0.25% in the test samples after sintering. Additionally, Zn-stearate was used in all mixtures as lubricant. The mixed powder mass was then compacted into dog bone tensile samples using a 100-ton capacity hydraulic press. The compaction was carried out under 700 MPa. The sintering of all samples was carried out in a control atmosphere tube furnace in argon. The sintering cycle applied to the samples involved: (i) heating to 1150°C at the heating rate of 5°C/min, (ii) holding at this temperature for 1 h, and (iii) cooling to room temperature at the cooling rate of 5°C/min. Sintering density values were obtained through water displacement using Archimedes' principle.

The tensile test was conducted at room temperature using a Schimadzu tensile testing machine at a crosshead speed of 1 mm · min^{-1}. The tensile testing machine was a constant rate of crosshead movement comprising essentially a fixed member carrying one grip and a movable member carrying a second grip. It was ensured that the tensile samples were perfectly aligned in the pulling direction and no slippage occurred at the grips. Triplicate samples were employed per run in order to correct for minor differences in experimental conditions.

Microstructural characterization was carried out on samples, which were mechanically polished using a standard metallographic procedure and etched with 2% Nital solution. The microstructure of the test samples was examined by using optical and scanning electron microscope (SEM). Also, the energy dispersive spectrometry (EDS) was used for elemental analysis of precipitate particles.

The grain size measurement was carried out using intercepts along a test line oriented at 45°. At least 500 grains, cut by intersecting line, were counted for each sample. After counting the grains, the total length of the intersecting lines was recorded and divided by the number of grains cut by intersecting line to determine mean lineal intercept grain size [9, 14]. The statistical errors associated with the measurement of the mean lineal intercept were calculated by using the statistical equation given by Blank and Gladman [15].

The volume fraction of ferrite and pearlite was calculated using a systematic point-counting method [16, 17]. According to the point-counting method, the grid points that fall in the ferrite and pearlite region are divided by the total number of grid points. When the grid points intersect the ferrite boundary, they are counted as half. Errors in the point-counting were also calculated by using the statistical equation developed by Gladman and Woodhead [18].

RESULTS AND DISCUSSION

Microstructure. Figure 2 shows light micrographs of Fe–0.25C (Alloy 1), Fe–0.25C–0.05V–0.05Ti (Alloy 2), Fe–0.25C–0.075V–0.075Ti (Alloy 3), and Fe–0.25C–0.1V–0.1Ti (Alloy 4) PM steel and microalloyed PM steels. All images were taken with the same magnification for direct visual comparison. It is seen that the primary microstructure of the PM steel and microalloyed PM steels are equiaxed ferrite and pearlite grains of different sizes. Table 1 also shows the relative density, the phase volume fractions, and the mean lineal intercept grain size under sintered condition.

It is observed that the grain size decreases with increasing the amount of Ti–V in the percentage of 0.1, 0.15, and 0.2 (Fig. 2, Table 1). The use of niobium, titanium, and vanadium as microalloying elements in steel for grain refinement is widespread, although the mechanisms by which this refinement is achieved are not completely understood. It is clear that the dissolution of these elements during reheating and prior to the hot deformation must be as predicted, if the desired effect is to be achieved for any given level of alloy addition. Microalloying elements can form carbides (stoichiometric or non-stoichiometric), nitrides, and carbonitrides. The type of precipitate formed

TABLE 1. Relative Density, Mean Lineal Intercept Grain Sizes, and Volume Fractions of Ferrite and Pearlite Phases in the PM Steel and Microalloyed PM Steels

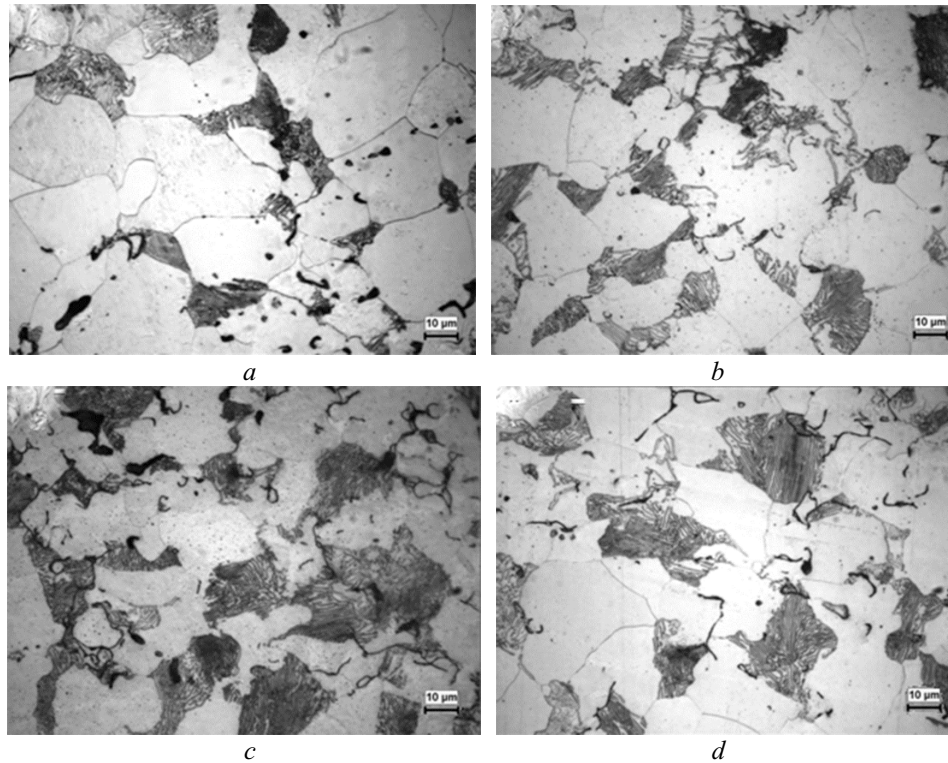| Alloy	Relative density, %	Grain size, μm	Ferrite, %	Pearlite, %
1	92	29.7	78.4	21.6
2	93	27.5	74.5	25.5
3	94	24.8	78.2	21.8
4	93	23.8	79.1	20.9

Fig. 2. Microstructures of sintered PM steel and microalloyed PM steels: Alloy 1 (*a*), Alloy 2 (*b*),
Alloy 3 (*c*), and Alloy 4 (*d*)

depends on the levels of carbon and nitrogen present and the levels of other alloy additives [19]. The Nb, Ti, and V microalloying elements precipitate in austenite (γ) as carbides, nitrides, and carbonitrides during hot rolling, and contribute to the mechanical properties of microalloyed steels via grain refinement, solid solution hardening, and precipitation hardening [20–23].

Ti forms nitride at a very high temperature and the nitride particles are very effective in controlling the grain growth of austenite. Ti levels in excess of that required to combine stoichiometrically with the results in combination with carbon, whose solubility is similar to NbC, thus preventing to austenite grains, which is giving rise to the fine ferrite grains [14]. Vanadium carbide, nitride, and carbonitride particles are known to pin austenite grain boundaries, and a fine ferrite grain structure develops during transformation [24]. It is evident that small quantities of vanadium in structural steels promote a significant refinement in the final ferrite microstructure, despite of enhancement of both the nucleation of grain boundary ferrite and intragranular nucleation of ferrite [25]. It is also considered that VC precipitates in ferrite together with TiC precipitates can prevent the ferrite grain growth and lead to a fine grain size in the final steel after sintering [26].

Mechanical Properties. Table 2 shows the mechanical properties of the PM steel and microalloyed PM steels. Increasing the titanium and vanadium content leads to an increase in YS and UTS of the PM steels. The elongation tends to improve with increasing Ti–V content. These changes would be expected as a result of the

TABLE 2. Mechanical Properties of Sintered PM Steel and Microalloyed PM Steels

Alloy	YS, MPa	UTS, MPa	Elongation, %
1	144	252	13
2	158	275	12
3	169	299	12
4	181	310	13

differences in precipitation distribution [27]. High strength and good toughness in microalloyed steels are achieved by a combination of microalloying and controlled rolling. The microalloying elements, such as niobium, titanium, and vanadium, facilitate the grain refinement through precipitation in austenite and contribute to the dispersion hardening through precipitation in ferrite during the austenite-to-ferrite transformation or after transformation [28].

The (Ti,V)(C,N) precipitates are formed during sintering and slow cooling from the sintering temperature. This leads to an increase in strength compared with the titanium- and vanadium-free alloy. It was established that the carbonitride precipitation plays an important role in Ti and V steels as for controlling the microstructure and mechanical properties. In fact, it has been reported that the ferrite grain size is influenced by the formation of fine precipitates during and after transformation, leading to very fine ferrite grains under particular processing condition [29].

The alloying elements have widely differing effects due to the different (i) solubility of their carbides and nitrides in both austenite and ferrite and (ii) precipitation kinetics. They increase in strength by grain refinement and precipitation effects, when sufficient carbon and nitrogen is present in the steel. The addition of alloying elements may restrict the austenite grain growth at the sintering temperature of 1150°C through the presence of precipitate particles (Fig. 3). It was observed that niobium is more effective in restricting grain growth than vanadium. Even titanium can be more effective due to the formation of titanium nitride [30, 31].

The solubility product of TiC and VC at 1150°C is $9.3 \cdot 10^{-3}$ and 1.1, respectively [32]. It is clear that the VC solubility is higher than that of TiC and, therefore, TiC should be present at the sintering temperature. However, due to higher VC solubility at 900°C, vanadium and carbon atoms should be in solid solution during sintering. It is believed that VC particles are formed during and after austenite-to-ferrite phase transformation, nucleating on the γ/α interface and in ferrite. One of the most effective mechanisms for increasing the strength of steels is: fine precipitates that form in ferrite during or after transformation. Vanadium is mostly used in microalloyed steels as precipitation strengthener. Vanadium is relatively soluble in austenite, but less soluble in ferrite and it transforms in the characteristic row formation [26].
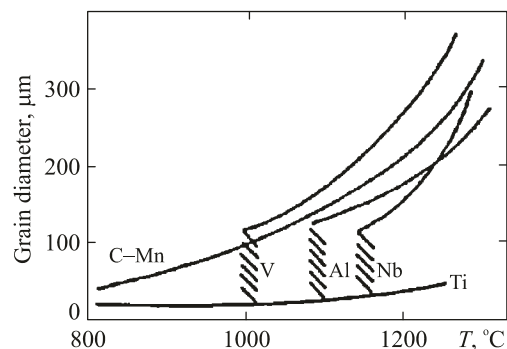

Fig. 3. Austenite grain growth in steels containing microalloy additives [29]

A higher solubility of vanadium compounds (and their solid solutions) ensures an adequate dissolution to give interphase precipitation, provided that the austenite-to-ferrite transformation conditions are suitable. This particular characteristic of vanadium has found its application not only in vanadium steel, but also as a supplement to the interphase precipitation strengthening in titanium steel [9].

According to stoichiometry, titanium (atomic weight 47.8) and vanadium (atomic weight 50.9) will combine with approximately one quarter its weight of carbon (atomic weight 12). Hence 0.1, 0.15, and 0.2 w/o Ti–V in microalloyed PM steel is not sufficient to remove all carbon from solid solution as TiC, VC, or (Ti,V)C. Depending on the sintering time, TiC, VC, and (Ti, V)C may be formed and there may be variable amount carbon in solid solution. During cooling from the sintering temperature, different amount of TiC, VC, and (Ti,V)C precipitates are formed leading to an increase in strength, compared with the Ti–V-free alloy (Alloy 1). Some reports [33, 34] show that, when the alloy contains several microalloyed elements, the precipitated carbides and

carbonitrides are more effective as a strengthener for having a complex composition of Ti, Nb, and V. The strength increment in microalloyed steel is mainly due to the precipitation hardening, and the effect of solid solution strengthening is low. The solid solution of microalloying atoms is negligible due to the lower concentration of microalloying atoms after formation of carbonitrides [35, 36]. In the present experimental study, the increase in strength of the microalloyed PM steel is due to the formation of TiC, VC, and (Ti, V)C.

In order to study the effect of precipitates and clusters on the strength of the microalloyed PM steel, it is necessary to calculate the value of σ_p which represent the strength obtained from precipitates and clusters in the microalloyed PM steel with different content of Ti–V. This was done using the equation given by Pickering and Gladman [37]:

$$\sigma_y = 54 + 17.4d^{-1/2} + \sigma_p, \tag{1}$$

where σ_y is the yield strength, d is the grain diameter (in mm), and σ_p is the strength obtained from precipitates and clusters. In this study, the σ_p values were calculated by using Eq. (1) for microalloyed PM steel. It was proposed that any difference between actual and predicted lower yield strength in the PM steel and microalloyed PM steels consist of a precipitation contribution σ_p, which includes an unknown contribution for clusters/solid solution strengthening. The calculation made has indicated that all the values of precipitation contribution (σ_p) vary from −10 MPa to 14 MPa for the Ti–V-free and Ti–V added microalloyed PM steels tensile-tested at room temperature. Table 3 provides the σ_p values.

TABLE 3. Structure Property Analyses of PM Steel and Microalloyed PM Steels Tensile-Tested at Room Temperature

Alloy	σ_o, MPa	Grain size, μm	$k_y d^{-1/2}$, MPa	σ_{total}, MPa	$\sigma_{y\ test}$, MPa	σ_p, MPa
1	54	29.7	100	154	144	−10
2	54	27.5	104	157	158	1
3	54	24.8	110	164	169	5
4	54	23.8	113	167	181	14

Different values for σ_p were observed in the PM steel and microalloyed PM steels (Table 3). It was found that the addition of Ti–V in the weight percentage of 0.1, 0.15, and 0.2 has increased the precipitation contribution (σ_p). This is due to the fine-dispersed grains and the precipitation contribution after sintering at 1150°C. Ti–V additives in the weight percentage of 0.1, 0.15, and 0.2 raised strength by precipitation strengthening and refining the structure. A similar effect was found in the study of Erden et al. [38]. They calculated the precipitation contribution (σ_p) in the Ti–C PM steel and observed an increase in σ_p when the Ti content raised to 0.1 and 0.2 w/o. However, the precipitation contribution in the Ti–V added PM steel was lower than that in the Ti added PM steel. This is due to the presence of V in the (Ti,V)C grain refining particles that reduces its content in VC precipitations after austenite-to-ferrite transformation. There are other reports on the reduction in strength of Ti–V steels in comparison with a corresponding V steel [8]. In the cast 0.10 V–0.023 Ti w/o steels [35], the formation of large TiN particles at high temperatures was considered to be responsible for the reduction in yield strength of 35 MPa, compared with a corresponding 0.092 V w/o steel, by removing nitrogen during formation of TiN particles and, therefore, decreasing the driving force for developing high density of the fine dispersion strengthening VN precipitates.

The pores are also expected to significantly affect the properties of the PM steel and microalloyed PM steels. The porosity of PM-produced components is usually in the range of 5–15%, depending on the compressibility of alloying powders, carbon content, and lubricant added [39–41]. The pores are the sites of potential crack initiation and can also guide and propagate the cracks through the material. According to Bergmark and Alzati [42], a strong microstructure may be obtained by incorporating small amounts of alloying elements to
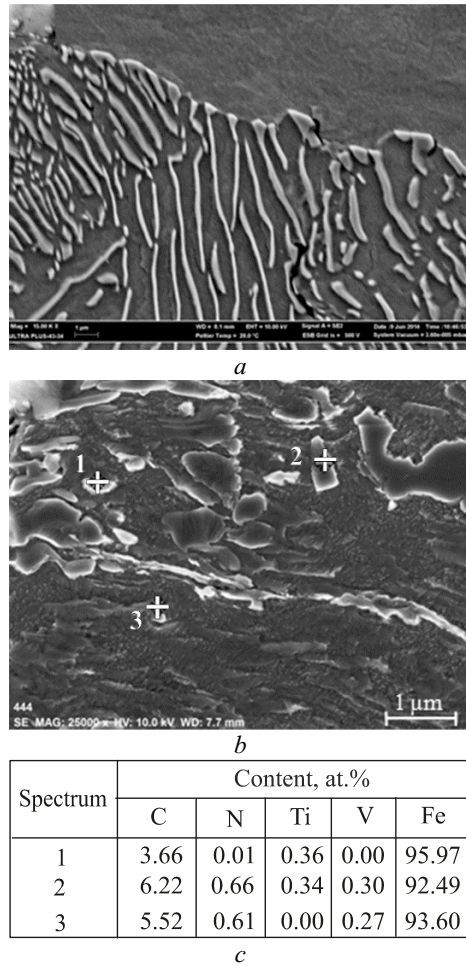
a

b

Spectrum	Content, at.%				
	C	N	Ti	V	Fe
1	3.66	0.01	0.36	0.00	95.97
2	6.22	0.66	0.34	0.30	92.49
3	5.52	0.61	0.00	0.27	93.60

c

Fig. 4. SEM images for Alloy 3: low magnification (*a*), high magnification (*b*), and corresponding EDS of the indicated points in Alloy 3 (*c*)

compensate for the microcracks formed by pores. In this study, the PM steel and microalloyed PM steels showed similar relative density (92 and 93%) for the as-sintered condition. This explains that an increase in strength is due to the presence of different amount of TiC, VC, and (Ti,V)C in the microalloyed PM steel.

Figure 4 shows EDS analysis with the spectrum points 1, 2, and 3 (precipitate particles) marked on the microstructure of Alloy 3 (Fe–0.25C–0.1V–0.1Ti). Point 1 contains Fe, C, and Ti, point 3 contains Fe, C, and V, but point 2 contains Fe, C, Ti, and V (Fig. 4). The presence of these elements indicates that TiC, VC, and (Ti,V)C occurred during sintering and/or after sintering. Siwecki et al. [43] investigated the precipitation behavior of a low carbon steel microalloyed with Ti and V. They found that adding Ti resulted in (Ti,V)N particles precipitating in austenite, which may have occurred by co-precipitation of VN nucleating on existing TiN particles. Chemical analysis indicated a compositional gradient which shows that the interior of the particles is rich in Ti, while the exterior is rich in V.

Figure 5 shows SEM images of the fracture surfaces of Alloys 1–4. Changes were observed in the fracture with respect to the size, shape, and depth of the microvoids. The image of Alloy 1 (Fig. 5*a*) demonstrated, that the fracture is purely ductile. This is evidenced by the presence of numerous dimples along with fine and rounded pores. Apparently, the mechanism of fracture is a formation of voids and coalescence. The cracks advanced largely by process of microvoid coalescence in the necks between the particles of the sintered steel. The microvoids nucleated and grew from microstructural defects, which resulted in the ductile type of the fracture. The microvoids nucleated at the re-localized strain discontinuities, such as those associated with *a priori* defects (pores, microcracks),
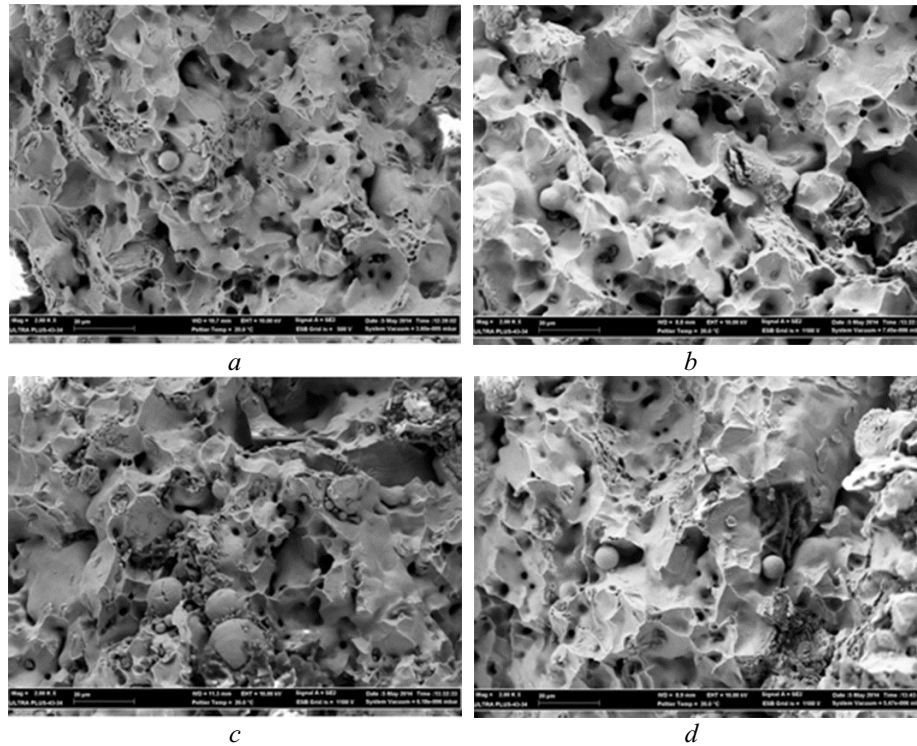
Fig. 5. Fracture surfaces of Alloy 1 (*a*), Alloy 2 (*b*), Alloy 3 (*c*), and Alloy 4 (*d*)

second phase particles, inclusions, grain boundaries, and dislocation pile-up. As the strain increases the microvoids grow, coalesce, and eventually form a continuous fracture surface; the crack extension in ductile fractures involves plastic deformation [44].

However, Alloy 2 (Fig. 5*b*), Alloy 3 (Fig. 5*c*), and Alloy 4 (Fig. 5*d*) showed dimples and cleavage facets indicating the mixed type of the fracture. In addition, the amount of fracture in Alloy 4 (Fig. 5*d*) is substantially higher than that in Alloy 1 (Fig. 5*a*), because Alloy 4 (Fig. 5*d*) contains more metal-to-metal fracture and less exposed pore surface area, compared with Alloy 1 (Fig. 5*a*). This is probably due to the strengthening effect of pearlite by alloying ferrite and reduction in the pearlite spacing. Adding V and Ti causes decrease in the transformation temperature and ferrite–pearlite forms at lower temperature resulting in finer ferrite and pearlite grains [45].

Large voids were also observed in the fracture surfaces (Alloys 2 and 3). These voids are indicative of the removal of TiC, VC, and (Ti,V)C particles through pulling-out under heavy tensile loading conditions. The pull-out of precipitate particles, such as TiC, VC, and (Ti,V)C on the crack faces, indicates a possible mechanism for the crack tip bridging in the microalloyed PM steels.

CONCLUSIONS

It has been established that the Ti–V addition in the weight percentage of 0.1, 0.15, and 0.2 increases the strength of steel through precipitation strengthening and refining the structure. It has been revealed that the precipitation of TiC, VC, and (Ti,V)C inhibits the grain growth during sintering. By inhibiting austenite grain growth, the precipitates have demonstrated a significant improvement in strength.

The EDS analysis has discovered that the particles of the microalloyed PM steel are rich in Ti, V, and Ti–V. The presence of these elements in the particles indicates that TiC, VC, and (Ti,V)C occurred during sintering and/or after sintering.

The analysis of the fracture surface has indicated that the PM steel with no microalloying additives exhibits ductile fracture. In the case of microalloyed PM steels, the fracture is ductile and brittle indicating the mixed type of

the fracture. This could be attributed to the pull-out of TiC, VC, and (Ti,V)C particles during deformation, indicating a possible mechanism for crack tip is bridging in microalloyed PM steels.

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