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Microstructural characterization and mechanical properties of microalloyed powder metallurgy steels

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ABSTRACT

The effects of Ti additions on the microstructures and mechanical properties of microalloyed powder metallurgy (PM) steels were investigated. The microstructure of the microalloyed PM steels was characterized with the help of optic microscope, SEM and EDS. Experimental results showed that Ti microalloyed steels can be produced by PM technology. The addition of Ti increases the strength in the sintered conditions. In addition, Ti limits grain growth during austenitization prior to cooling. By limiting austenite grain growth, the precipitates result in significant improvement in strength.

1. Introduction

The volatility of the cost of metals has led to the development of new alloy systems for PM steels. In particular, PM steels have utilized copper, nickel, and molybdenum to compete with wrought grades. Recently, due to cost constraints and availability, PM steels have also included chromium and manganese as alloying elements. These material systems are categorized as alloy steels since significant levels of these elements are required to effect changes in the mechanical properties. Microalloyed steels are defined as steels containing small amounts of niobium, vanadium, or titanium, generally at levels between 0.05% and 0.20%. Their specific effects may be influenced by other alloying additions such as aluminum, boron, or indeed any of the other more conventional alloying elements used in steel manufacture. The effects of the microalloying elements are also strongly influenced by thermal and thermomechanical treatments [1,2].

The addition of microalloying elements offers an important cost-effective approach to obtain a good combination of excellent toughness and strength through grain size control and precipitation hardening. This is a result of the formation of carbonitrides, which leads to both precipitation strengthening and grain refinement. These beneficial properties have been achieved by a careful control of chemical composition and by adopting suitably controlled thermo-mechanical processes which are different in PM than in conventional wrought-steel processing utilizing forging and rolling [1,3–5].

Powder metallurgy (PM) process is a versatile and efficient route for producing components with combinations of various alloying elements. These alloying elements significantly improve the hardenability by shifting the transformation curves to the longer transformation time. Subsequent heat treatment results in enhancing the mechanical properties of the ferrous alloys [6–8]. To ensure similar properties as those of quenched and tempered steels, Si and Mn are added to increase the strength of the microalloyed steels. Further increase in the strength is achieved by the addition of microalloying elements, through the precipitation of carbonitrides [9]. Titanium microalloying technology is an effective method to produce hot rolled high strength steel through precipitation hardening of titanium carbides [10]. Ti in microalloyed steels is more efficient for strengthening than V and Nb due to lower solubility, which increases the equilibrium fraction of the carbides substantially compared with the same amount of carbide-forming elements [11]. Also, TiC has sparked considerable

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interest because of its good wettability with Fe, high hardness and low density compared to the other components. Hard particles distributed in the matrix cause an increase in strength, stiffness, wear resistance and decreased density [12–14].

In the present study, titanium was added to several microalloyed PM steels. Mechanical properties were measured and microstructures characterized in the sintered condition to assess the role of precipitation strengthening and grain refinement.

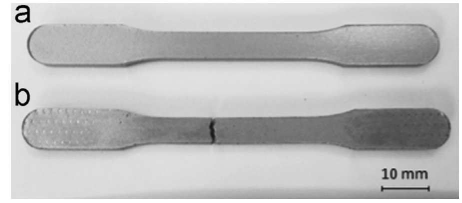

Fig. 1. General view of tensile test specimen sintered at 1150 °C for 1 h; (a) before fracture and (b) after fracture.

2. Materials and experimental procedure

Fe and Ti powders were purchased from Aldrich in mean particle sizes of 74 and 12 μm, respectively. Fe–0.25C, Fe–0.25C–0.1Ti, Fe–0.25C–0.15Ti and Fe–0.25C–0.2 Ti powders were prepared by mixing for 1 h in an industrial conical mixer. Graphite additions were 0.45% to reach carbon concentration of 0.25% in the test pieces after sintering. Additionally, Zn-stearat was used in all the mixes as the lubricant. Dog-bone tensile specimens were compacted at a pressure of 700 MPa using a hydraulic press of 100 tons capacity. Sintering of all the samples was carried out in a controlled atmosphere tube furnace in an argon atmosphere. The sintering cycle applied to the samples involves heating to 1150 °C at a rate of 5 °C/min and holding at that temperature for 1 h and then cooling to room temperature at a rate of 5 °C/min. Sintering density values were obtained through the water displacement Archimedes method.

Tensile test was carried out at room temperature using a Schimadzu tensile testing machine at a crosshead speed of 1 mm min^{-1}. Triplicate samples were employed per run in order to correct for minor differences in the experimental conditions. Fig. 1 shows the tensile test specimen used in this study.

The examination of steel microstructures was carried out using an optic and scanning electron microscope (SEM). Energy dispersive spectrometry (EDS) was used to provide elemental analysis on precipitate particles. The specimens were polished according to standard metallographic methods. Etching times were closely monitored to prevent overetching of the alloys due to the fineness of their microstructure. The microstructures were examined in a Nikon ECLIPSE L150 type microscope capable of magnifications between 50× and 1000×. The grain size measurement was carried out using intercepts along a test line oriented at 45°. At least 500 grains cut by the intersecting line were counted for each sample.

3. Results and discussion

The variation in mechanical properties in PM steel and microalloyed PM steels can be explained in terms of microstructure obtained cooling after sintering. Fig. 2 shows the evaluation of the microstructure for Fe–0.25C (Alloy 1), Fe–0.25C–0.1Ti (Alloy 2), Fe–0.25C–0.15Ti (Alloy 3) and Fe–0.25C–0.2Ti (Alloy 4) PM steel and microalloyed PM steels under sintered conditions. It is seen that the optical microstructure of PM steel and microalloyed PM steels consists of ferrite and pearlite structures. Table 1 also shows relative density, the phase volume fractions and mean linear intercept grain sizes under sintered condition.

As can be seen, the addition of Ti in the percentages of 0.1, 0.15 or 0.2 led to finer grain sizes. A major benefit of microalloying is the restriction of grain growth during austenitizing. If fine precipitates exist during the austenitizing step, the growth of grains is restricted, leading to a finer grain size after quenching [1,15]. Ti forms a nitride at a very high temperature and the nitride particles are very effective in controlling grain growth of the austenite. The extremely low solubility of TiN is such that the addition of even modest microalloying levels can induce precipitation of a nitrogen-rich Ti(CN) in the liquid steel. Ti levels in excess of that required to combine stoichiometrically with the results in combination with the carbon, the solubility of which is similar to NbC, thus prevent the austenite grains which gives rise to the fine ferrite grains [2].

Table 2 illustrates the mechanical properties of PM steel and microalloyed PM steels by showing yield strength (YS), ultimate tensile strength (UTS), percentage elongation and percentage yield point elongation. In the sintered condition there is general increase in YS and UTS when titanium is present. During sintering and slow cooling from sintering temperature TiC(N) precipitates form, and these lead to an increase in strength compared with the strength of titanium-free alloy. The role of microalloy precipitates depends on the temperature at which it forms in relation to the transformation temperature of steel. The temperature at which simple nitrides and carbides form in relation to transformation temperatures as steel cools during and after processing is illustrated in Fig. 3. These data are based on equilibrium conditions and give an indication of the relative temperatures at which the various compounds form. In practice, the rate at which steel cools will however determine the actual temperature at which precipitates and transformation occur. The higher the cooling rate the lower the temperature at which the precipitates form. The rate of cooling can, in fact, determine whether a precipitate forms in austenite or ferrite [16].

From the solubility product data, it is known that the solubility of TiN is higher than that of TiC and therefore TiN should be present at the austenitizing temperature. In the present experimental work, the amount of soluble Ti atoms at 1150 °C was calculated by a simple quadratic Eq. (1) given by Gladman [2] and also solubility product Eq. (2) given by Narita [17] for TiC from which the amount of Ti and C in solid solution at 1150 °C was found to be 0.04 and 0.236, respectively. This soluble Ti will precipitate as TiC in ferrite depending on the cooling rate. The solubility data of Irvine et al. [18] gives much lower values of 0.029 and 0.233 for soluble titanium and carbon, respectively. This would suggest that more titanium present as TiC (71% compared with 60%) would be expected if the Irvine data were applied to titanium microalloyed steel, but nevertheless implies that some titanium and carbon remain in the solution at a sintering temperature of 1150 °C.

$$\mathrm{Ti}_{TiC}=\frac{A_{Ti}[((\mathrm{C}_T+\mathrm{Ti}_T A_C)/A_{Ti})-(((\mathrm{C}_T+\mathrm{Ti}_T A_C)/A_{Ti})^2-(4A_C(\mathrm{Ti}_T\mathrm{C}_T-k_s)/A_{Ti}))^{1/2}]}{2A_C} \tag{1}$$

$$\log\ [\mathrm{Ti}\%]_\gamma[\mathrm{C}\%]_\gamma=-\frac{10475}{T}+5.33 \tag{2}$$

According to stoichiometry, titanium (atomic weight 47.8) will combine with approximately one quarter its weight of carbon (atomic weight 12). Hence, 0.1, 0.15 or 0.2% Ti in microalloyed PM steel is not sufficient to remove all the carbon from the solid solution as TiC. Depending on the sintering time used, TiC may be formed and there may be variable amount of carbon in the solid