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Development of mechanical performance of 12YWT steel nanocomposite by addition of zirconium and tantalum

Roohollah Rahmanifard [a, *], Hasan Farhangi [b], Abdul Javad Novinrooz [c]

[a] School of New Technologies, Iran University of Science and Technology, Tehran, Iran
[b] School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran
[c] Islamic Azad University, Takestan Branch, Department of Science, Physics Group, Takestan, Iran

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ABSTRACT

The 12Cr–3W–0.4Ti–0.25Y_2O_3 (12YWT) Oxide dispersion strengthened ferritic steel shows a good strength up to 600 °C, while degrades considerably beyond that. In addition, this steel undergoes a week ductile to brittle transition temperature. Studies were made to improve of both strength and DBTT. Hence, the mechanically alloyed powders containing different contents of Zr (0.5–1.5%) and Ta (0.05–0.15) were extruded at 850 °C and mechanical properties of these steel nanocomposites were investigated. Mechanical analysis studies indicated that the addition of zirconium element can improve mechanical properties. For example, the presence of 1.5% zirconium in 12YWT resulted in an unprecedented value of 2100 MPa for tensile strength and improved ductile to brittle transition temperature of −27 °C. In contrast of zirconium, increase of tantalum did not change remarkably tensile strength of 12YWT. However, Charpy impact test results presented that tantalum have an important role in improved ductile to brittle transition temperature so that this parameter changed from 13 °C to −44 °C at presence of 0.15% Ta.

1. Introduction

Oxide dispersion strengthened (ODS) reduced activation ferritic steels have been developed as a promising candidate structural material for application in new generation nuclear power plants. The frequently used route to produce the ODS materials is mechanical alloying followed by the hot deformation process [1–3].

In these materials, oxide nano-clusters (NCs) formed during consolidation process would allow a substantial increase of the operating temperature up to 650 °C or even more and also a superior resistance to irradiation induced damage depending on size and thermal stability of them [4–6]. Complex NCs of Y–Ti–O and Y–Zr–O have higher binding energy with respect to Y–O NCs resulting in higher thermal stability with finer size. Furthermore, oxide bonding length for Zr is shorter than that for Ti which elucidates stronger interaction between Zr and Y and consequently finer NCs [7]. Dou and Kimura [8] found that utilizing Zr in SOC-9 ODS steel, the mean size and number density of nano-oxide particles in grains were considerably smaller and much higher, respectively. Capability of Zr to form finer complex oxide particles of Y–Ti–Zr–O with respect to conventional Y–Ti–O particles in 12YWT was confirmed in our previous study [9]. The reduced particle size and increased number density of dispersoids along with proper particle distribution can enhance effective obstacles against migration of grain boundaries and as a result a decrease in grain size which may improve tensile and impact property of ODS steel. There is only a report on improvement of tensile properties of Fe–16Cr–2W–0.5Ti–0.4Y_2O_3–4Al affected by Zr [10], hence, the further efforts are required to achieve an understanding about how Zr may affect the mechanical properties of ODS steels.

In contrast to outstanding high temperature strength, weak ductility and poor impact toughness are main drawbacks that limit the application of ODS steels. Since the oxide particles are the origin for crack nucleation [11,12], hence it seems even above-mentioned attempts to develop the finer dispersoids with higher number density may deteriorate the impact energy which invoke the further investigations. However, there are various approaches to improve the toughness of materials. The crystalline grain size and chemical composition are two major parameters that can strongly affect impact properties [12]. Achieving the ultrafine grained structure can be a beneficial solution that has been investigated elsewhere [13]. About Incorporation of alloying element, the

* Corresponding author.
E-mail address: rahmanifard@iust.ac.ir (R. Rahmanifard).

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studies had been mostly concentrated on the matrix materials. Ni and Mn were known as toughening elements in steels [12]. But since both these elements produce radioactive isotopes with half-lived longer than Fe, therefore it was avoided to use in reduced activated ferritic steels [14]. It has been reported that Ta has a positive effect on both ductile to brittle transition temperature (DBTT) and strength of reduced activation steels [15–18]. Even though Ta like Nb is known as a strong carbide former and can impede the grain growth leading to the improved strength, it has been observed that the major part of Ta remains in solid solution [15]. It has been expressed that sufficient Ta available in solid solution increases the cleavage stress and causes superior impact properties [16–18]. Since there were not any investigation on impact properties of ODS materials influenced by Ta, it is merited to evaluate whether this element is profitable. Therefore in this study are assayed effects of Zr and Ta on mechanical behavior of 12YWT ODS ferritic steel.

2. Experimental procedure

The 12YWT–ODS steel with the chemical composition of Fe–12.3Cr–3.0W–0.4Ti–0.25Y_2O_3 (wt%) was prepared by mechanical alloying, using high-purity metal powders of Fe, Cr, W and Ti in size about 10 μm and Y_2O_3 particles sized between 20 and 30 nm. In order to investigate effects of Zr and Ta on the mechanical properties of 12YWT, the various amounts of high purity of Ta and Zr powders in size of micrometer in the range of 0.05–0.15 wt% and 0.5–1.5 wt% respectively, were used. The powder mixture was mechanically blended for 2 h before milling. The mechanical alloying was performed in a high purity Ar (99.9999 wt%) atmosphere with optimized condition given in Ref. [19] using an attritor ball mill. The milled powders were then sealed into a mild steel can, degassed at 400 °C under vacuum of 10^{-4} mbar and consolidated into a bar by hot extrusion at 850 °C. Tensile tests were carried out using sheet specimens according to ASTM-E8M with a gage section 1.5 × 3 mm^2 and gauge length of 13 mm which were machined from the extruded bars in a direction parallel to the hot-extrusion direction. The tests were performed at several temperatures from room temperature to 800 °C at strain rate of 6×10^{-4} s^{-1}. In order to obtain a reliable result, the average of three specimen was reported. Charpy impact tests were performed on V-notch KLST specimens (3 × 4 × 27 mm^3) according to DIN 50115 using an instrumented Charpy impact machine with an energy capacity of 50 J at temperatures ranging between −150 °C and 300 °C. KLST specimens were also machined parallel to the extrusion axis (L–T orientation). The ductile-to-brittle transition temperature (DBTT) was determined through fitting the data by the following function [20]:

$$E = \frac{a}{2}\left[1 + \tanh\left(\frac{T - T_0}{c}\right)\right] + \frac{b}{2}\left[1 - \tanh\left(\frac{T - T_0}{c}\right)\right] \quad (1)$$

where, E is Charpy energy, a is upper shelf energy, b is lower shelf energy, c is a measure of the temperature range over which the transitional behavior occurs and T_0 is DBTT. It is necessary to note that tensile and Charpy impact tests were performed three times for each testing temperature. The fracture surfaces of specimens were also evaluated using Scanning Electron Microscopy (SEM) by a Philips XL30. Transmission Electron Microscopy (TEM) by a Philips EM208S was utilized to characterize the microstructure of materials and determination of size distribution of dispersoids. The density of the samples was also measured using Archimedes' principle according to ISO 2738 standard.

3. Results and discussion

The chemical composition of the extruded ODS ferritic steels used in this work are presented in Table 1. These materials contains a relatively high oxygen amount which originates from the surface of elemental powder particles and the mechanical alloying atmosphere, and a relatively high carbon amount that comes from the grinding steel media. As a long milling time of 80 h used in this study allows a long exposing time of particles against milling atmosphere and grinding media are promoted these milling-induced impurities. However, almost no significant differences in impurity contents were measured for different specimens. Only the specimens containing higher Zr show a slight increase in oxygen content which may be attributed to the very strong affinity of Zr for oxygen. This can result in a higher number density of dispersoids as observed in microstructural investigations [9] and probably affects the mechanical properties of these materials.

Typical microstructure of 12YWT alloyed with Zr and Ta is shown in Fig. 1. With regard Fig. 1(a), the microstructure mostly consists of nano-sized equiaxed grains with no grain alignment along the deformation axis whereby non uniformity of mechanical properties in different directions which is a main challenge for common extruded materials can be improved. Moreover, bright and dark filed TEM images in Fig. 1(a) and (b) clearly show an uniform distribution of nano-sized dispersoids throughout the matrix which is accompanied some coarser grain boundary precipitates. Detailed information relating to microstructural characteristics of these ODS steels can be found in Ref. [9].

The stress–strain curves shown in Fig. 2 describe how the Zr and Ta affect the tensile properties of 12YWT at room temperature. As observed, addition of these elements to the base alloy may increase the strength of this alloy which can be induced by the increased density of dispersoids and a noteworthy decrease in crystalline grain size according to microstructural observation reported in Ref. [9]. Thus, it is expected that the presence of these elements develop the elastic zone at expense of plastic one. A salient point in Fig. 2 is a significant increase of yield and tensile strengths in 1.5% Zr-containing alloy. According to ref [13] the mean yield strength for base alloy is 1390 MPa while by adding 0.5% and 1.5% Zr according to Fig. 3(a), this parameter reaches to 1780 and 1880 MPa at room temperature, respectively showing an increase of 28% and 35%. Zirconium has a similar effect on the tensile strength. The presence of 0.5% and 1.5% zirconium as observed in Fig. 3(b) change the tensile strength of 12YWT from 1470 MPa to 1970 and 2100 MPa, respectively indicating an increase of 34% and 43%. It is necessary to state that the highest value reported by researchers on reduced activation ODS steels is related to 14YWT obtained by Kim and et al. [21]. They succeeded to attain the strength of 1750 MPa using hot extruding at 850 °C followed by a multi-step hot rolling for 40% thickness reduction. A comparison between the strength reported for 14YWT and obtained for 12YWT containing 1.5% Zr, shows an increase of 20% in tensile strength.

Fig. 2 shows that the addition of tantalum from 0.05% to 0.15% doesn't remarkably improve the strength of 12YWT. Our previous study on microstructural properties of 12YWT revealed that the grain size was not influenced by Ta [9]. Therefore, a minor increase in strength may be attributed to the presence of Zr in Ta-containing alloys.

According to Fig. 2, 12YWT steel nanocomposite sustains a strain softening in plastic deformation zone. The more detail about this phenomena were explored in Ref. [13]. This figure indicates that Zr and Ta intensify the rate of strain softening. At the same time, the phenomenon of sudden decrease in stress which is observable at low temperatures is also promoted with the presence of these elements. The increase of crack initiation sites such as dispersoids