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Microstructural and mechanical characterization of Cu-0.8 wt.%Y

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Abstract
Dispersion strengthened Cu-0.8 wt.%Y has been produced by a powder metallurgy route and subsequent consolidation by hot isostatic pressing at 1123 K and 172 MPa. A fully dense alloy has been obtained that exhibits a microstructure characterized by equiaxed grains with sizes ranging from 0.5 to 50 µm. Yttrium-rich particles with an average size of 0.52 µm have been observed inside the grains and decorating the grain boundaries. As expected, the tensile tests carried out from room temperature to 773 K have revealed that both the YS and the UTS decrease with increasing temperature. This alloy exhibits better tensile properties and microhardness than OFHC Cu. This improvement is attributed to the presence of the Y-rich particles.

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1. Introduction

An important component in the future fusion reactors is the cooling system, whose task will be to extract heat from the blanket and divertor [1,2]. High temperature He cooled systems and low temperature water-cooled systems are the two cooling concepts that have been conceived for the fusion reactors [3-5]. The water-cooled designs with Cu-alloys as heat sinks are being considered for the cooling system of ITER [6].

Pure copper has a very high thermal conductivity but its mechanical strength is relatively low. Furthermore, the service life of pure copper is limited because of its high creep, swelling rate and irradiation hardening. There are different methods for enhancing the strength of pure copper, such as cold working, grain refinement, solid solution, precipitation hardening and dispersion strengthening [7]. The enhanced mechanical properties of CuCrZr with respect to pure copper are due to precipitation induced by aging [8]. CuCrZr alloys with composition Cu-(0.5-1.2%)Cr-(0.03-0.3%)Zr (wt%) are the primary candidates for heat sink materials in ITER due to its good weldability and high fracture toughness at high temperature. However, under neutron irradiation these CuCrZr alloys present a loss of ductility at low temperature and worsening of the strength at elevated temperatures. Above 450 °C the dissolution of the precipitates is activated and the degradation of the mechanical properties occurs. These drawbacks limit the operating temperature window of CuCrZr [7,9].

Keywords:
• Copper
• Cu-Y alloys
• High isostatic pressing
• Powder metallurgy
• Reinforced materials

The oxide dispersion strengthened (ODS) alloy Cu-0.5 wt.% Al2O3 (Glidcop Al25) has been one of the Cu alloys considered for ITER [10,11]. In the case of Cu alloys, oxide dispersion strengthening presents advantages with respect to solution and precipitation hardening. First, there are no risks that the precipitates dissolve in the matrix. And second, the dispersion of nano-particles in the copper matrix prevents recrystallization, increases the resistance to softening at high temperatures and does not affect to the thermal conductivity [12-14].

Y2O3 as alternative dispersoid to Al2O3 for ODS Cu has been proposed to enhance the mechanical properties and thermal conductivity of a matrix of Cu [15,16]. According previous studies reported elsewhere [17], it appears that a Y2O3 content as high as 1 wt.% might promote an appropriate dispersion.

In this work a Cu-0.8 wt.%Y alloy has been fabricated via a powder metallurgy route using pre-alloyed powders. A study of the microstructure and mechanical properties of the alloy is reported.

2. Experimental

Powder with nominal composition Cu-0.8 wt.%Y starting from 99.9% pure Cu and 99% pure Y was produced by vacuum induction melting and subsequently atomization in high purity Ar gas. The particle size distribution of the powder was evaluated by laser diffraction. The powder, with particle size <120 µm, was encapsulated into a steel can that was tight sealed after degassing at 573 K for 24 h in vacuum. Then, the powder consolidation was accomplished by hot isostatic pressing (HIP) for 2 h at 1123 K and 179 MPa. A fully dense alloy with a density of 8.895 ± 0.015 g/cm³ instead of the theoretical value of 8.889 g/cm³ was obtained. The...
density measurements were carried out in a He ultrapycnometer and the theoretical density calculated applying the mixture rule. The oxygen contents of the powder and consolidated alloy determined by atom absorption resulted in 0.070 wt.% and 0.038 wt.%, respectively. The reduction of the oxygen content in the consolidated material is attributed to the degassing process that removes part of the volatile components incorporated to the powder.

Both the alloyed powder and the consolidated alloy were characterized by X-ray diffraction (XRD). The XRD patterns were analyzed by the Rietveld method using the Fullprof software [18]. The microstructure of the atomized Cu-0.8Y powder and the consolidated alloy were studied by optical microscopy, scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDS). For analyzing the powder particles, the powder samples were embedded in an epoxy resin and, after polishing, the surface etched for revealing the grains. The etchant used for revealing the grain structure was a 6 vol.% HNO₃ solution in methanol. The grain size distributions were obtained from optical microscope images. The mean grain size in the powder was obtained from an area based count method. In the case of the consolidated material the mean size was determined by the linear intercept method. Vickers microhardness measurements were performed at room temperature on polished samples applying a load of 0.098 N during 20 seconds. The tensile tests were accomplished through the temperature range 293-773 K on flat samples 1 mm thick, 3 mm width with a gauge length of 15 mm. The tests were carried out in a Shimadzu testing machine under flowing Ar at a constant strain of 1.11 x 10⁻⁴ s⁻¹.

3. Results and discussion

3.1. Powder characterization

The volume particle distribution of the Cu-0.8Y powder is shown in Fig. 1(a). This distribution is described by a logarithmic-normal function with sizes of 19, 28 and 63 µm for volume fractions of 30, 50 and 90%, respectively. The microstructure of the powder particles is shown in Fig. 2(a). It consists of spherical particles whose grain size depends on the size of the particle so that the grain size increases with particle size. This result is in good agreement with the fact that in the atomization process cooling rate for the smaller particles is considerably faster than for the large ones resulting in smaller grain sizes. The dependence of the mean grain size on the particle size is shown in Fig. 1(b). A more detailed SEM image of the powder microstructure is shown in Fig. 2(b). EDS analyses revealed the Y enrichment of the grain boundary neighborhood and the absence Y or other impurities inside the grains. This is attributed to the presence of some Cu-Y intermetallic compound [19].

The XRD pattern of the powder particles is shown in Fig. 3. Besides the peaks corresponding to the Cu, a peak associated to the intermetallic compound Cu₄₂Y is also observed. This result is in good agreement with the EDS analyses and the Cu-Y phase diagram.

3.2. Consolidated alloy characterization

3.2.1. Microstructure

Fig. 4 shows the microstructure of the consolidated alloy. It consists of equiaxed grains with sizes ranging from 0.5 to 50 µm. The numerical distribution of grain sizes is depicted in Fig. 5. The morphology and size of the initial powder particles are identified after consolidation. No changes are apparent in their morphology and size but grain growth is found in many of them (see Fig. 4).

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The X-ray diffraction pattern for the consolidated alloy, depicted in Fig. 3, does not exhibit any peak associated with secondary phases in spite of the microstructural evidences and the presence of Cu$_2$Y in the atomized powder. This can be attributed to the transformation of Cu$_2$Y into other intermetallic phases during the HIP at 1123 K treatment [19]. The fraction of these phases would be below the detection limit of the X-ray diffractometer, i.e., ~1%. TEM experiments could verify the presence of these new Cu-Y intermetallic phases.

The SEM and EDS analyses showed the presence of Y-rich particles uniformly distributed inside the matrix grains besides at the grain boundaries, in contrast with that observed for the alloyed powder. The size distribution of these particles is depicted in Fig. 6. It appears to correspond to a lognormal distribution with an average size of 0.92 ± 0.05 µm.

**3.2.2. Mechanical properties**

The microhardness value for the consolidated material resulted in 660 ± 20 MPa, which is remarkably higher than the value of 393 MPa reported for OFHC Cu (grain size ~20 µm) [20]. This result indicates that the presence of yttrium-rich particles in the copper matrix induces a considerable hardening.

Fig. 7 shows representative true stress–strain curves of the consolidated alloy. The thermal evolution of the yield strength, YS, ultimate tensile strength, UTS and plastic uniform strain at UTS, $e_{yf5}$, are displayed in Fig. 8. As expected for a metallic material, the mechanical strength of Cu-0.8Y decreases on increasing temperature. Both, YS and UTS decrease linearly increasing temperature. The strain hardening ratio defined as the ratio UTS/YS also experiments a continuous reduction going down from 3.6 at room temperature to 2.1 at 773 K. It should be noted that the uniform strain remains practically constant up to 573 K but increase steeply at higher temperatures.

The present Cu-0.8Y alloy in the as-HIP condition exhibits tensile properties better than annealed OFHC Cu in the investigated temperature range but lower than the CuCrZr ITER grade alloy [21–23].

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SEM images of the fracture surfaces at room temperature are shown in Fig. 9. A dimple pattern showing ductile fracture is clearly visible. From the dimple aspect it is induced that the fracture is caused by nucleation and growth of voids around the yttrium-rich particles. Cracks appear around the grain boundaries indicating that the fracture is intergranular.

4. Conclusions

A fully dense Cu-0.8Y alloy was produced by HIP exhibiting a microstructure that consists of a Cu matrix of equiaxed grains with sizes ranging from 0.5 to 50 µm and Y-rich particles, with a mean size of 0.92 µm, uniformly distributed inside the grains and along the grain boundaries. These particles have been attributed to different intermetallic Cu-Y compounds. The microhardness and tensile properties of the Cu-0.8Y alloy are superior to the ones for annealed OFHC Cu. This strengthening is attributed to the presence of the Y-rich particles. It is expected that further grain and particle refinement through thermo-mechanical processing treatments could enhance the mechanical properties.

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