Development of Y$_2$O$_3$ Dispersion-Strengthened Copper Alloy by Sol-Gel Method

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Abstract: In this study, oxide dispersion-strengthened Cu alloy with a Y$_2$O$_3$ content of 1 wt.% was fabricated through citric acid sol-gel synthesis and spark plasma sintering (SPS). The citric acid sol-gel method provides molecular mixing for the preparation of precursor powders, which produces nanoscale and uniformly distributed Y$_2$O$_3$ particles in an ultrafine-grained Cu matrix. The effects of nanoscale Y$_2$O$_3$ particles on the microstructure, mechanical properties and thermal conductivity of the Cu-1wt.%Y$_2$O$_3$ alloy were investigated. The average grain size of the Cu-1wt.%Y$_2$O$_3$ alloy is 0.42 µm, while the average particle size of Y$_2$O$_3$ is 16.4 nm. The unique microstructure provides excellent mechanical properties with a tensile strength of 372 MPa and a total elongation of 6.4%. After annealing at 800 °C for 1 h, the strength of the alloy does not decrease obviously, showing excellent thermal stability. The thermal conductivity of Cu-1wt.%Y$_2$O$_3$ alloy is about 308 Wm$^{-1}$K$^{-1}$ at room temperature and it decreases with increasing temperature. The refined grain size, high strength and excellent thermal stability of Cu-1wt.%Y$_2$O$_3$ alloys can be ascribed to the pinning effects of nanoscale Y$_2$O$_3$ particles dispersed in the Cu matrix. The Cu-Y$_2$O$_3$ alloys with high strength and high thermal conductivity have potential applications in high thermal load components of fusion reactors.

Keywords: copper; sol-gel; mechanical properties; thermal stability; thermal conductivity

1. Introduction

Copper alloys are the main candidate materials for the heat sink of the water-cooled target of a divertor in nuclear fusion reactors, due to its high thermal conductivity, strength and good radiation resistance [1–5]. Pure copper has a very high thermal conductivity but its strength is relatively low especially at high temperatures. Furthermore, the service life of pure copper is limited because of its high creep, swelling rate and irradiation hardening [6,7]. Therefore, it is necessary to develop copper alloys with both high strength and good thermal conductivity.

CuCrZr alloys are the primary candidates for heat sink materials in the International Thermonuclear Experimental Reactor (ITER) due to its high thermal conductivity and high strength at medium and low temperature [8–10]. However, long-term thermal exposure of CuCrZr alloy to excessive temperature (>400 °C) leads to loss of strength due to over-ageing where the precipitates undergo Ostwald ripening losing their effective resistance as a barrier against dislocation glide [11]. Furthermore, neutron irradiation will cause degradation of thermal conductivity and mechanical properties of the alloy [12]. These drawbacks limit the operative temperature window of CuCrZr alloys. Developing a high-performance copper alloy to provide the best combination of excellent mechanical strength and good thermal conductivity at high temperatures is the main trend. Dispersion-strengthened copper alloys are an effective method to improve the high temperature strength of Cu-based...
materials. Highly stable ceramic particles (such as Al₂O₃ [11,13], TiC [14,15], NbC [16] and Y₂O₃ [17–19]) dispersed in the copper matrix can effectively hinder the motion of dislocations and migration of the grain boundary and extend the operating temperature range of copper alloys [20]. Among them, the Y₂O₃ dispersion-strengthened Cu alloys attract intense attention due to their excellent mechanical strength with higher thermal conductivity. The Cu-0.46wt.%Y₂O₃ alloy (Glidcop Al25) has been one of the Cu alloys considered for ITER [5,21].

Recently, Y₂O₃ dispersion-strengthened copper alloys have attracted increased attention. Compared with alumina, Y₂O₃ is relatively more suitable because of the low solubility of element Y in copper (less than 0.05 wt.% at 300 °C), which helps to maintain a high thermal conductivity [17]. In addition, the high thermodynamic stability of Y₂O₃ enhances the coarsening resistance at high temperatures [22]. Many efforts have been devoted to develop Y₂O₃ dispersion-strengthened copper alloys by Mechanical Alloying (MA) methods [6,7,17,18,23–26]. For example, G. Carro et al. [6] investigated the microstructural and mechanical properties of Cu-0.8wt.%Y produced by a powder metallurgy route and subsequent consolidation by hot isostatic pressing. The resultant equiaxed grain size distribution ranged from 0.5 µm to 50 µm, which greatly weakens the effect of fine grain strengthening. S.M.S. Aghamiri et al. successfully prepared a Cu-0.42wt.%Y₂O₃ alloy with comparable strength to the Glidcop-Al25 alloy by adding stearic acid as a process control agent and using MA and spark plasma sintering (SPS) [23]. Bing MA et al. explored the influence of Cu-Y compound content on the microstructure of Cu-Y₂O₃ alloys synthesized by MA and HIP process [26]. However, Y₂O₃ particles are prone to aggregation and growing up in the MA synthesized Cu-Y₂O₃ alloys, and impurities are easily introduced in the MA process. As the mechanical properties of dispersion-strengthened alloys are closely linked to the size and distribution of dispersoids, the segregation and large size of Y₂O₃ in copper alloys would inevitably reduce the mechanical properties.

In this work, a citric acid sol-gel method [27] involving molecular level mixing was adopted to synthesize nanoscale Y₂O₃ particles homogeneously dispersed copper powders, and ultrafine-grained Cu-1wt.%Y₂O₃ alloy with high strength, high thermal conductivity and excellent thermal stability was obtained through spark plasma sintering (SPS) from sol-gel synthesized powders. The influence of Y₂O₃ nanoparticles on the mechanical properties, microstructure and thermal conductivity of the alloys was investigated and the corresponding mechanisms were discussed.

2. Materials and Methods

Cu-1wt.%Y₂O₃ alloy powders were synthesized using a citric acid sol-gel method. Raw materials for the synthesis of Cu-1wt.%Y₂O₃ powders are listed in Table 1. The specific experimental process is as follows: C₆H₁₂O₇·H₂O was firstly dissolved in deionized water to create an acidic environment, then Cu(NO₃)₂·3H₂O and Y(N₂O₉)·6H₂O were added into the solution. Citric acid is employed as chelating agent, which can provide the mixing of cations at the molecular level in the sol-gel process. The solution was heated at 80 °C with continuous stirring. An appropriate amount of polyethylene glycol (PEG, molecular weight 20,000) was added as surfactant after stirring at 80 °C for 2 h. The temperature was maintained at 80 °C with continuous stirring to remove excess water until a gel formed. The resulting gel was dried at 140 °C for 12 h and then was calcined at 550 °C for 6 h in air to remove the organic compounds, leading to a black mixture of CuO and Y₂O₃ powders. The calcined oxide powders were further ground to crush large particles, and subsequently, the oxides mixture was reduced in flowing hydrogen at 400 °C for 1 h, resulting in Cu-1wt.%Y₂O₃ powders.

The sol-gel synthesized Cu-1wt.%Y₂O₃ powders were consolidated at 900 °C for 5 min under a pressure of 50 MPa using an SPS method to obtain bulk samples and the specific process can be seen in the literature [28]. The size of the sintered samples was 30 mm in diameter and about 3 mm in thickness. The densities of the samples were determined by the Archimedes principle using distilled water as the immersion liquid. The theoretical
density of Cu-1wt.%Y$_2$O$_3$ alloy is 8.85 g/cm$^3$ according to the rule of mixtures and the relative density of the sintered samples is about 97.6%.

Table 1. Raw materials for the synthesis of Cu-1wt.%Y$_2$O$_3$ powders.

| Raw Materials               | Molecular Formula           | Purity       |
|----------------------------|----------------------------|--------------|
| Citric acid monohydrate    | C$_6$H$_8$O$_7$·H$_2$O      | ≥99.5%       |
| Copper nitrate trihydrate  | Cu(NO$_3$)$_2$·3H$_2$O      | ≥99%         |
| Yttrium nitrate hexahydrate| YN$_3$O$_9$·6H$_2$O         | ≥99.99%      |
| Polyethylene glycol        | HO(CH$_2$CH$_2$O)$_n$H      | -            |

The samples for the tensile tests were machined to a gauge dimension of about 5 × 1.5 × 0.75 mm$^3$ using an electrical discharge machine and slightly polished. The tensile tests were carried out at room temperature (RT) with an Instron 5967 testing machine at a constant speed of 0.3 mm/min (corresponding to a nominal strain rate of 1 × 10$^{-3}$ s$^{-1}$) in air.

The thermal conductivity ($\gamma$) was calculated from the thermal diffusivity ($\alpha$), density ($\rho$) and specific heat ($C_p$) according to the relation: $\gamma = \alpha C_p \rho$. The $\alpha$ and $C_p$ was determined using the laser flash diffusivity system (LFA457 Micro flash, NETZSCH). The sample was cut into cylinders with a diameter of about 12.6 mm and a thickness of about 2.5 mm and polished smooth with sandpaper. The thermal conductivity was measured at the Engineering Materials Science Experiment Center of University of Science and Technology of China (USTC, Hefei, China).

The sol-gel synthesized oxides precursor and reduced Cu-1wt.%Y$_2$O$_3$ powders were characterized by X-ray diffraction (XRD, X'Pert) with Cu Kα radiation. The morphology of the powders and tensile fracture of bulk samples was characterized by a field emission scanning electron microscope (FE-SEM, SU8020). Electron backscatter diffraction pattern (EBSD) mappings were collected using a Zeiss SIGMA field emission scanning electron microscope (SEM) equipped with a CRYSTAL detector (Oxford Instruments, Oxfordshire, UK) and the acceleration voltage was 13 kV. A misorientation angle of $\theta > 10^\circ$ was used to distinguish the grain boundaries. The EBSD images were slightly filtered through the software (HKL Tango) to eliminate the influences of second phase particles on Cu grain observations. Microstructure characterization was carried out by a transmission electron microscopy (TEM, Tecnai G2 F20) at 200 kV to determine the size and distribution of Y$_2$O$_3$ particles in Cu-1wt.%Y$_2$O$_3$ alloys. Energy-dispersive X-ray spectroscopy (EDS, INCA) installed in the TEM was used for elemental analysis. The TEM samples were prepared by cutting flaked samples with diameters greater than 3 mm from the SPS sintered sample. First, the flakes were ground to about 50 µm in thickness. Subsequently, a Gatan 659 disk punch was used to cut a 3-mm-diameter disk from the thin wafer and then fixed on a molybdenum ring with an outer diameter of 3 mm. Then the specimen was transferred to Gatan 623 and further thinned to about 30 µm. Finally, the precision ion milling system (Gatan model 691) was used to polish the disk specimens at 4.3 keV to obtain electron transparency in the middle part. Note that the final angle and voltage of ion thinning for the TEM samples preparation in this study were 3$^\circ$ and 3.2 keV, respectively.

### 3. Results and Discussion

#### 3.1. Characteristics of Powders

Figure 1 shows the XRD patterns of the sol-gel synthesized precursors before and after hydrogen reduction. The powder composition before hydrogen reduction is mainly composed of CuO and Y$_2$O$_3$ through software (Highscore plus 3.0) analysis. The XRD diffraction peaks at 35.55°, 38.67° and 48.84° correspond to the (11-1), (111) and (20-2) plane of CuO (PDF: 98-004-3179). After hydrogen reduction at 400 °C for 1 h, the XRD peaks at 43.33°, 50.46° and 74.15° corresponding to the (111), (002) and (022) plane of Cu (PDF: 98-062-7113) indicates that the CuO powders were completely reduced. The XRD diffraction peaks at 20.49°, 29.14° and 33.77° correspond to the (112), (222) and (004) plane.
of Y$_2$O$_3$ (PDF: 98-001-6394). However, no diffraction peak of yttrium oxide was found in XRD. This may be due to the low content and poor crystallinity of yttrium oxide, which will be further analyzed later in the powder TEM. Figure 2a shows the SEM images of the sol-gel synthesized precursor powders, consisting of CuO and Y$_2$O$_3$. The particle size distribution of the precursor powders is 20~200 nm with an average particle size of about 80 nm. After hydrogen reduction at 400 °C for 1 h, Cu particles grow up and sintering necks are formed between some Cu particles, as shown in Figure 2b,c. Figure 2d shows the high-magnification image of the rectangular region marked with a red dotted line in Figure 2c. Some nanoscale particles were evenly distributed on the surface and inside of the copper particles and the uniform distribution of these nanoparticles in copper powders is the prerequisite for obtaining high performance Cu-Y$_2$O$_3$ alloys.

![Figure 1](image1.png)

Figure 1. XRD patterns of sol-gel synthesized oxides and the powders hydrogen-reduction at 400 °C for 1 h.

![Figure 2](image2.png)

Figure 2. SEM images of (a) sol-gel synthesized precursor powders, (b,c) hydrogen reduced Cu-1wt.%Y$_2$O$_3$ powders at 400 °C for 1 h, and (d) magnification of the region marked with red dotted line in (c).

Figure 3a,b shows the TEM images of the powders reduced at 400 °C for 1 h. Most of the particles are spherical, and some sintering necks were formed between the particles. Figure 3c shows the EDS results of Figure 3a, and the results show that there are
three elements Cu, O and Y in the powders. Selected area electron diffraction (SAED) of Figure 3a is shown in Figure 3d; the result shows that the powders are composed of Cu and Y$_2$O$_3$. Elemental mapping analysis was performed on the rectangular region in Figure 3b to determine the composition of these particles, as shown in Figure 3e. The results show that most of these particles are Cu, while the distribution of Y and O elements and SAED results further confirm the existence of Y$_2$O$_3$. Figure 3f shows the high resolution TEM (HRTEM) image of the particles indicated by the rectangle in Figure 3e. The measured interplanar spacings of the two particles are 0.306 nm and 0.433 nm, respectively, corresponding to the (222) and (112) crystal planes of Y$_2$O$_3$ (PDF: 98-001-6394).

![Figure 3. TEM images of hydrogen-reduced Cu-1wt.%Y$_2$O$_3$ powders at 400 °C for 1h, (a,b) TEM bright field images, (c) EDS results of the reduced powders, (d) SAED patterns of (a), (e) element mapping of the rectangular area in (b), and (f) HRTEM of Y$_2$O$_3$ particles indicated by the rectangle in (e).](image)

### 3.2. Microstructure Characterization

The mechanical properties of dispersion-strengthened materials is closely linked to the grain structure and the size and distribution of the strengthening phases. Figure 4a,d shows the inverse pole figure (IPF) maps of as-sintered Cu-1wt.%Y$_2$O$_3$ alloy and the corresponding grain size distribution. The majority of Cu grains in the as-sintered Cu-1wt.%Y$_2$O$_3$ alloys are in the submicron range with an average grain size of 0.42 μm. Compared with the Cu-5vol.%Y$_2$O$_3$ alloy (average grain size 1.15 μm) prepared by mechanical alloying [24], the grain size of the Cu-1wt.%Y$_2$O$_3$ alloy is obviously refined. After annealing at 800 °C, the grain growth in the Cu-1wt.%Y$_2$O$_3$ alloy is not obvious with an average grain size of 0.45 μm, as shown in Figure 4b,c. As the annealing temperature increases to 900 °C, the average grain size of the Cu-1wt.%Y$_2$O$_3$ alloy increases to 0.54 μm, and the annealed sample consist of two different types of grains: larger recrystallized grains and smaller submicron grains, as shown in Figure 4c,f. This phenomenon is attributed to the recrystallization of copper during high-temperature annealing. The recrystallized grains nucleate from the grain boundary and then many small submicron grains grow into recrystallized grains. Meanwhile, the Y$_2$O$_3$ particles distributed in the Cu matrix can effectively inhibit the growth of the grain and many grains are still smaller than 0.5 μm in size even after 900 °C annealing. Overall, the recrystallization temperature of Cu-1wt.%Y$_2$O$_3$ alloy samples is significantly higher than that of the Cu samples, and Y$_2$O$_3$ particles plays a significant role in the improvement of grain refinement and thermal stability.
The measured interplanar distance of the Y$_2$O$_3$ particle and Cu matrix is 0.306 nm and 0.209 nm, respectively, corresponding to the values of the Y$_2$O$_3$ (222) and Cu (111) planes from the Powder Diffraction File (PDF) card of 98-001-6394 and 98-006-4699. In addition, the local area of particle distribution is analyzed through elemental distribution mapping, as shown in Figure 5e,f. Through the elemental distribution mapping of Cu, Y and O and analysis results of HR-TEM, it can be determined that the particles uniformly distributed in Cu matrix are Y$_2$O$_3$ particles. In fact, the uniform distribution and small size are attributed to the Cu-1wt.%Y$_2$O$_3$ alloy powders prepared using the citric acid sol-gel method and the short sintering time by SPS, which effectively reduces the number and size of Y$_2$O$_3$ particles at the grain boundaries. These Y$_2$O$_3$ nanoparticles with a high melting point and high hardness greatly improve the strength and thermal stability of the alloy.

Figure 6 shows HAADF-STEM images and Y$_2$O$_3$ particles size distribution of the Cu-1wt.%Y$_2$O$_3$ samples annealed at 800 °C and 900 °C. After annealing at 800 °C, the average particle size of Y$_2$O$_3$ is 17.0 nm and the size and distribution of Y$_2$O$_3$ particles do not change significantly. After annealing at 900 °C, the grain size increased slightly to 17.6 nm. This phenomenon is consistent with the change of mechanical properties, which further explains the high thermal stability of the copper alloy.

3.3. Mechanical Properties

Figure 7 shows the engineering stress-strain curves of the Cu-1wt.%Y$_2$O$_3$ alloy and pure Cu annealed at 800 °C and 900 °C. The ultimate tensile strength (UTS) and total elongation (TE) of the alloy at RT are 572 MPa and 6.4%, respectively (Figure 7a). The strength
is significantly improved compared with the pure Cu sample prepared by the same process (Figure 7b). In addition, compared with the Y2O3 dispersion strengthened copper alloy prepared by MA, the strength of the alloy has been greatly increased. For example, Zhou et al. reported Cu-5vol%Y2O3 alloys prepared by mechanical alloying, high temperature heat treatment and powder compact extrusion, with UTS and TE of 389 MPa and 8.9%, respectively [24]. The UTS of the present Cu-1wt.%Y2O3 alloys is similar to that of the Cu-60wt.%W composite prepared using the melt-infiltration method [30]. Compared with precipitation-strengthened CuCrZr alloy and dispersion-strengthened CuAl-25 alloy, the strength of the Cu-1wt.%Y2O3 alloy is obviously improved, while the ductility is decreased [31]. The low ductility of the Cu-1wt.%Y2O3 alloy may arise from the relatively high porosity (2.4%) and the particles distributed at the grain boundaries. The pores and intergranular Y2O3 particles would act as stress concentrators and cause localized plastic deformation [30]. After annealing at 800 °C, the UTS of Cu-1wt.%Y2O3 alloys had no obvious change with a high value of 563 MPa and the TE was 5.2%. After annealing at 900 °C, the UTS decreased to 532 MPa, which may be caused by partial recrystallization at this temperature. In a word, Cu-1wt.%Y2O3 alloys prepared using sol-gel method have a high strength and good thermal stability.

![Figure 5. DF-STEM images (a,b), Y2O3 particles size distribution (c), HRTEM images of Cu and Y2O3 phase interface (d), and elemental distribution mapping (e,f) of Cu-1wt.%Y2O3 alloy.](image)

![Figure 6. HAADF-STEM images and Y2O3 particles size distribution after annealing at different temperatures: (a-c) 800 °C annealing; (d-f) 900 °C annealing.](image)
These defects would scatter electrons and therefore reduced the thermal conductivity [28]. The thermal conductivity of the Cu-1wt.%Y$_2$O$_3$ alloy was measured at different temperatures from RT to 800 °C. The thermal conductivity of copper alloys is one of the most important parameters that determines whether they can be used as heat sink material for future fusion reactors. However, there are few mentions in the literature about the thermal conductivity of Y$_2$O$_3$ dispersion strengthened copper alloy. Figure 9 shows the thermal conductivity of the Cu-1wt.%Y$_2$O$_3$ alloy was measured at different temperatures from RT to 800 °C. The thermal conductivity of the Cu-1wt.%Y$_2$O$_3$ alloy at room temperature is 308 Wm$^{-1}$K$^{-1}$, which is lower than that of pure copper. The decrease in thermal conductivity after Y$_2$O$_3$ addition can be ascribed to the relatively lower thermal conductivity of Y$_2$O$_3$, and more defects such as the grain boundaries and Cu/Y$_2$O$_3$ phase interfaces than in pure Cu. These defects would scatter electrons and therefore reduced the thermal conductivity [28]. The thermal conductivity of the alloy decreases with the increasing temperature. Even so, the thermal conductivity at 800 °C of the Cu-1wt.%Y$_2$O$_3$ alloy is above 250 Wm$^{-1}$K$^{-1}$, which is still superior to the value of the W-Cu composite [32].
Figure 9. Thermal conductivity of Cu-1wt.%Y$_2$O$_3$ alloy at different temperatures.

4. Conclusions

Nanostructured Cu-1wt.%Y$_2$O$_3$ alloy with high strength, high thermal conductivity and good thermal stability was fabricated by sol-gel synthesis and the SPS method. The sol-gel process involving molecular mixing can be used to prepare nanoscale Y$_2$O$_3$ homogeneously dispersed copper powders. The average particle size of Y$_2$O$_3$ in Cu-1wt.%Y$_2$O$_3$ alloy is only 16.4 nm, which can effectively refine grains and improve the strength and thermal stability of the material by pinning dislocations and grain boundaries. The average grain size of a Cu-1wt.%Y$_2$O$_3$ alloy is about 0.42 μm. The tensile strength and thermal conductivity of the nanostructured Cu-1wt.%Y$_2$O$_3$ is 572 MPa and 308 Wm$^{-1}$K$^{-1}$, respectively. The strength and microstructure of the alloy do not change significantly after annealing at 800 °C, indicating excellent thermal stability. This work provides a promising method for fabricating high performance nanoparticle dispersion-strengthened Cu alloys.

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