



Thermophysical properties of Cu-ZrO₂ composites as potential thermal barrier materials for a DEMO W-monoblock divertor

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ABSTRACT

DEMO fusion reactor divertor is expected to extract a heat flux of about 10 MW/m². One of the most promising concept design for it is the W-monoblock, which should be connected to a CuCrZr or an advanced Cu ODS alloy pipe passing through the W component. Since the optimum operating temperature windows for W and existing Cu alloys are far away from overlapping, a suited interface is needed to keep the adjacent materials as close as possible to their respective temperature operating windows. The interface material should therefore have a low enough thermal conductivity to act as a thermal barrier and a thermal expansion coefficient suited to protect the W-pipe joint from stresses induced by the different thermo-mechanical properties of W and Cu-alloys. As interface materials we have considered Cu-ZrO₂ composites produced by powder metallurgy route. Such materials can be realized in an unexpected large compositional range (up to at least 90% ZrO₂ volume concentration) and be easily further joined to both W and Cu-alloys by an electrical field assisted technology. We analyse their microstructure and thermo-physical properties both as single materials and included in W-thermal barrier-CuCrZr 3-layers systems in comparison to those of previously produced Cu-ased composites and commercially available Cu foams.

1. Introduction

DEMO [1] is a key point in the European fusion roadmap, being a fusion reactor able to deliver energy to the grid. The materials used for it should withstand both high heat fluxes and intense neutron irradiation for long times, ranging between 2 and 5 years [2]. In the case of its divertor, an expected heat flux of about 10–20 MW/m² should be extracted. Thus, a full W armour is considered as the most viable option, while the following heat sink part will be most likely constructed from Cu or ODS Cu alloys pipes [3], similar to the ITER full-W divertor design [4]. W has a high melting point, a high sputtering threshold and low tritium retention [5,6], which are desired properties for a plasma facing material, but W also has a rather high ductile-brittle transition temperature (DBTT), around 300–400 °C as measured by impact bending tests performed on unnotched specimens with dimensions of 3 × 3 × 27 mm³ [7]. This value sets the lowest limit of its operating temperature window, while the upper limit can be derived from recrystallization constraints at about 1200 °C. In fact, the optimum operating temperature for W is considered to be around 800–1000 °C, taking into account recovery considerations [8–11]. To remove the large heat flux from the divertor, materials with high thermal

conductivity are needed in the heatsink. Such materials will support the W components and will be also exposed to neutron irradiation. Thus high strength and good irradiation behaviour are also required properties. One possible candidate is the Cu precipitate strengthened (PS) alloy CuCrZr [12] or similar oxide dispersion strengthened (ODS) Cu alloys. For CuCrZr alloys the temperature operating window is between 180 °C and about 300 °C [13,14]. The upper limit value is considerably lower as the W optimal operation temperature. To keep both materials in their respective operating temperature ranges an interface material is needed. Such a material should slow down the heat transfer from the W hot part to the CuCrZr cool part, acting like a thermal barrier (TB) [15–17]. In the same time, taking into account the large difference between the W and Cu thermal expansion coefficients (CTE), it is important that the interface material will have a CTE value in-between, thus decreasing the thermal generated stresses [18]. To solve this particular problem, different interface materials have been suggested [19–23], but these have also thermal conductivities between Cu and W values and therefore are not acting like a proper thermal barrier. Our previous work [17] has shown that some Cu-based composites can be efficient thermal barrier materials, while mitigating in the same time the effects of the different thermal expansion coefficients of W and Cu

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(CuCrZr). In the W-monoblock divertor concept [14] the W armour is formed as a rectangular prism and a CuCrZr pipe is passing through the W component. In this case the thermal barrier will create a functional joint between the armour and heatsink and should be thinner as 1 mm [16,18,24]. These means, taking into account the large temperature difference between W and CuCrZr optimum operating temperatures, low thermal conductivities values for the interface material, e.g. around 15 W/m/K and an elastic modulus of around 1 GPa for a 0.5 mm layer thickness [16]. For these reasons, in the present work, we have considered Cu-ZrO₂ composites as potential candidates for a DEMO divertor thermal barrier material. ZrO₂ is a ceramic material having a high melting temperature, with one of the lowest thermal conductivities for such oxides, and a good compatibility with Cu. For the obtained materials we investigate the sintering behaviour, the resulting morphology and the relevant thermo-physical properties. The possible joining of the thermal barrier with W and CuCrZr is also evaluated.

2. Experimental

The Cu-ZrO₂ thermal barrier materials have been prepared using micrometric Cu powders (average particle size, APS = 1 μm) and nanometric ZrO₂ powders (APS = 20 nm) provided by US Research Nanomaterials, Inc. The powders have been mixed in Ar protective atmosphere in various volume proportions, ranging from 10 to 90% for the dispersed materials, using a planetary ball mill at low speed (50 rpm). The homogenized compositions have been sintered in graphite moulds using a spark plasma sintering (SPS) equipment at different temperatures for 5 min. Since the real temperature inside the moulds can be only approximated in the following the SPS average control temperature will be used to discriminate between samples. The maximum sintering temperature which can be used was determined by the limit at which Cu starts to melt. The thermal barriers have been connected to W and CuCrZr using the same SPS equipment.

The samples' morphology was checked by SEM using a microscope equipped with backscattering detector (BSD), used to evaluate the distribution of the elements in the sample. The thermal transport properties have been investigated using a Netzsch LFA 457 Microflash up to 1000 °C and the expansion coefficients have been determined in the same temperature range using a Netzsch 402C dilatometer. The electrical resistivity was measured up to 800 °C using a SBA 458 Netzsch equipment, in a 4 contact point configuration. The LFA equipment allows the direct measurement of the thermal diffusivity, while the specific heat of materials, can be determined by a differential method using a reference sample. The thermal conductivity is calculated by $\lambda = \alpha \times \rho \times C_p$, with ρ the density and α the diffusivity of the material. In the case of a 2-layer sample, assuming the thermal properties are known for each material layer, the thermal contact resistance can be determined and used to evaluate the joint quality. We have used this method on components made by joining a thermal barrier material to a W and a CuCrZr plate, respectively. For all samples the density was measured by Archimedes method using a high resolution balance.

3. Results and discussion

3.1. Sintering of Cu-ZrO₂ specimens

The SPS technology is based on a multitude of effects, resulting in high heating and cooling rates and very short overall processing time. This is because the heating arises from high intensity dc current pulses trains applied to the graphite mould and sample at the same time. While the current flowing through the mould provides a convection heating from outside, the current flowing through samples is on one side heating the metallic materials by Joule effect and on the other side creating small electric discharges at the imperfect grains connections [25]. This phenomena is assumed to be responsible for mass transfer and an enhanced sintering. For ceramic samples, the high intensity

current flows on the surface of the grains and again is supposed to produce discharges and mass transfer at the imperfect grain connections [26]. In the case of metal-ceramic composites, the phenomenology is more complicated and difficult to quantify. If the volume fraction of metal is high, the current will flow through the metal increasing its temperature. Thus if the melting temperature of the metal is considerably lower than the melting temperature of the ceramic, the metal part will be overheated resulting in a catastrophic melt of the sample. On the other hand, if the ceramic volume fraction is high, one can expect that above a threshold ceramic concentration the sample will be in the best case only partially sintered (e.g. metal-metal connections and eventually metal-ceramic connections). This limitation was expected to occur also in the case of Cu-ZrO₂ composites. To increase the composition range we have chosen to use nanometric oxide powders which should allow a better dispersion in the Cu matrix. As in the case of Cu-Al₂O₃ or Cu-Y₂O₃ composites [17], a maximum oxide concentration of about 50% volume was expected. Surprisingly, in the present case, we have been able to consolidate specimens with ZrO₂ volume concentrations up to 90%. Up to a volume concentration of about 80% ZrO₂ a SPS average temperature of 830 °C can be used to obtain well consolidated materials. Higher temperatures can be used at lower ZrO₂ content (like e.g. 930 °C, up to 70%). Increased temperatures result in a small amount of melted Cu emerging from the samples.

An answer for the unexpected good sintering behaviour can be given by morphological investigations of the samples. In Fig. 1 the densities of the produced samples are displayed as a function of the ZrO₂ volume content. The relative densities were calculated using the measured values in relation with the theoretical densities calculated using the direct mixing rule. The low temperature sintering curve exhibits an unusual trend, slightly increasing at lower concentrations (up to about 40% ZrO₂ volume), followed by a decrease toward a minimum and thereafter an increase to values close to 98% relative density. Going to the maximum concentration results in a small amount of Cu lost from the sample and therefore also a decrease in density (because the Cu lost during SPS was not included in the theoretical density calculation). Higher temperature sintering is able to provide higher densities up to about 60% ZrO₂ volume concentration, then the relative density starts to decrease and at 80% we have already melted Cu lost from the sample. For low temperature sintering, the increase of relative density at lower concentrations can be ascribed to better sintering. The SEM images presented in Fig. 2a) and b) show that ZrO₂ powders are agglomerated between the larger Cu grains. Increasing the ZrO₂ content and thus decreasing the Cu amount in the SPS implies an increase of the local temperature of Cu and as a result a better sintering and densities. However going to higher ZrO₂ content (above ~50% volume) breaks the continuity of Cu in many areas and this leaves large ZrO₂ clusters with a subsequent lower quality sintering of the samples and lower

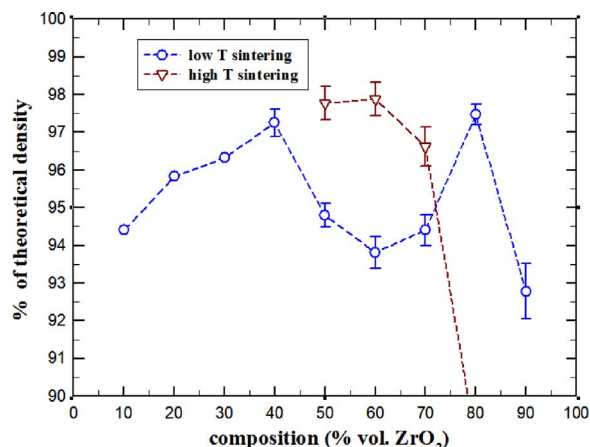


Fig. 1. Relative densities of Cu-ZrO₂ composites sintered at 830 °C and 930 °C.

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