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Effect of grain size on thermal residual stresses and damage in sintered chromium–alumina composites: Measurement and modeling

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ABSTRACT

In this paper we present the results of experimental measurements and numerical modeling of the effect of particle size on the residual thermal stresses arising in sintered metal–matrix composites after cooling down from the fabrication temperature. On example of novel Cr(Re)/Al_2O_3 composites processed by (i) spark plasma sintering and (ii) hot pressing the residual thermal stresses are measured by neutron diffraction technique and determined by a FEM model based on micro-CT scans of the material microstructure. Then numerical model of microcracking induced by residual stresses is applied to predict the effective Young modulus of the damaged composite. Comparison of the numerical results with the measured data of the residual stresses and Young's modulus is presented and fairly good agreement is noted.

1. Introduction

Metal matrix composites (MMC) are important structural materials in many industrial applications (e.g. automotive, aerospace and rail transport) because of their high specific stiffness and strength, enhanced wear and fatigue resistance and superior properties in high temperature regimes [1]. However, the residual stresses arising in processing of bulk MMCs can adversely affect the performance of structural elements made of these materials. MMCs can be produced by sintering under pressure or without pressure application, wherein the temperature (depending on the constituent materials used) can reach up to 2000 °C. During the cooling from the fabrication temperature thermal stresses arise within the composite due to different thermal expansion coefficients of the phase materials, variable cooling speed at different locations inside the material, or irregular shape of the pores that can lead to concentration of thermal stress [2].

The most popular experimental methods to measure residual stresses are: (i) X-ray diffraction [3,4] and (ii) neutron diffraction [5–7]. The basic principles behind both methods are the same and involve the measurement of lattice elastic deformation and distortion (i.e. macro and micro strains) from the displacement and broadening of the diffraction peak. However, X-rays are good for near-surface stress measurements since their depth of penetration for metals like copper or aluminum is 30–100 μm. For bulk composites, neutron diffraction method is more effective for in-depth average stress measurement due to the high penetrating power of neutrons in most engineering materials [8].

The literature on numerical modeling of thermal residual stress by FEM in metal–ceramic composites is abundant (e.g. [9–12]). A numerical model of the 1st order residual stresses and damage, making use of micro-computed tomography images of material microstructure in the preparation of FE mesh was recently proposed by the authors [13,14]. For complex reinforcement shapes in composites this approach seems to be an efficient way of FE discretization. An inspiring research problem with potential impact on the residual stresses has emerged in [13] due to a large difference in the size of metal (Cr) and ceramic (Al_2O_3) particles in the commercial powders used.

The objective of this paper is to investigate experimentally by neutron diffraction and numerically using the model proposed in [13] the grain size effect on the thermal residual stresses and their influence on the overall Young modulus of novel Cr(Re)/Al_2O_3 bulk composites sintered by HP and SPS techniques. The admixture of rhenium to the composite under consideration was motivated by certain high temperature applications in the transport industry.

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2. Processing and characterization of composites

The composite material was processed by two widely used powder metallurgy techniques: hot pressing (HP) and spark plasma sintering (SPS), [15–18]. Two kinds of commercial chromium powders were used with an average grain size of 40 μm for HP sintering and 5 μm for SPS. The aluminum oxide and the rhenium powders used for SPS and HP were the same and had average grain size of 2–5 μm for alumina and 4.2 μm for rhenium, respectively. It is to be emphasized that adding rhenium to Cr/Al_2O_3 composite is an original element of this research work in the processing part. It was motivated by material requirements for structural elements in automotive applications operating in demanding in-service conditions, which are combinations of high temperature, corrosive environment and high pressure. A prior analysis of the phase diagram of the Cr–Re system and the thermodynamics of the process had shown that at the applied sintering temperature a dissolution of rhenium in the chromium matrix is to be expected. As a consequence a new solid solution will be formed enhancing the strength of the composite material under investigation.

The grain size of the starting powders was examined using Clemex TV image analyzing system. The fact that different sintering techniques (HP vs. SPS) were used is noted but it was not of primary effect on the residual stresses. The main drivers for these stresses are different CTE's of the metal and ceramic phases.

The powder mixtures were prepared using a planetary ball mill (Pulverisette 5) with balls of 10 mm diameter. The mixing parameters were as follows: rotational speed $\omega = 200$ rpm, mixing time $t = 4$ h. The powder mixtures were densified under pressure of 30 MPa and temperature of 1300 °C for SPS (SPS HP 5 apparatus, FCT, Germany) and 1400 °C (Cr/Al_2O_3) and 1450 °C (Cr/Re/Al_2O_3) for HP (Thermal Technology Astro Press, USA), respectively. More details on the processing conditions can be found in [13,14].

The densities of the sintered composite samples were measured using the hydrostatic method. Taking into account the volume fractions of the phase materials, theoretical densities of the composites were defined using the density of aluminum oxide $\rho_{Al2O3} = 3.97$ g/cm^3, chromium $\rho_{Cr} = 7.19$ g/cm^3 and rhenium $\rho_{Re} = 21$ g/cm^3. The measured and theoretical densities of the composites are given in Table 1. It can be seen from Table 1 that densities achieved for samples sintered with chromium of 5 μm average grain size are higher than those of 40 μm. SEM observations revealed a very good homogeneity and no visible pores in the microstructure of the composite made with 5 μm Cr gains (Fig. 1a). For Cr/Al_2O_3 composites made with 40 μm Cr powder the porosity was detected mainly in the ceramic phase. Another observation was that due to large grain size difference between the constituent phases, the grains of alumina oxide tend to form clusters surrounding the big chromium grains.

It is to be mentioned that TEM and XRD analyses in [19] have shown that rhenium tends to form solid solutions with chromium in the composites under consideration.

The three point bending test was used for measurement of Young's modulus. The measurement procedure followed that of DIN EN 843 [20,21]. The average values of Young modulus calculated from five tests are shown in Table 1. The addition of rhenium leads to an increase of Young's modulus. It can be seen that the composites 75Cr/25Al_2O_3 and 75Cr/25Al_2O_3 + 5Re with coarser Cr powder have lower density that those with fine Cr grains. Also, the respective Young's moduli reveal the same tendency (247 GPa vs. 280 GPa and 278 GPa vs. 310 GPa). The higher density of samples with 5 μm Cr grains can be traced back to the better homogeneity achieved with the fine grain chromium powder, Fig. 1a.

A comment on the processing by the hot pressing (HP) vs. spark plasma sintering (SPS) technique seems in order when reporting on the measured results for density (porosity) and Young's modulus of the fabricated composites. The porosity is lower for the SPS processed samples, being of the order of 1% or less (cf. the first two lines in Table 1), than for the HP samples, where it is about 3% as shown by the last two lines in Table 1. As the elastic properties of materials are naturally affected by the porosity, a similar effect can be noted for Young's modulus of the SPS made samples as compared with the HP ones (cf. Table 1). However, since the chromium powder used in the SPS process was much finer than that in the HP (5 vs. 40 μm), the resulting porosities of the Cr(Re)/Al_2O_3 composites should be traced back to this grain size difference rather than SPS vs. HP sintering techniques, as these mainly differ by the way sample is heated and also by the duration of the process. In fact, the particle sizes of constituent materials have a major effect on the consolidation of the composite powder in a die and, thus, on the porosity of sintered sample. Smaller particles increase the total surface area in the compact, hence the driving force for densification is increased leading to lower porosity of the composites made with smaller grains, the sintering technique SPS vs. HP being of less important effect.

In order to investigate thermal residual stresses in the obtained Cr/Al_2O_3 composites, neutron diffraction experiments were performed on the DIANE diffractometer at the Léon Brillouin Laboratory in Saclay (France) with a fixed neutron wavelength $\lambda = 2.9$ Å, by means of which the Bragg peaks corresponding to the reflection (1 1 0) for Cr (scattering angle $2\theta \approx 90°$) and (1 1 3) for Al_2O_3 (scattering angle $2\theta \approx 87.5°$) could be investigated.

The investigated gauge volume was $1 \times 1 \times 1$ mm^3, determined by 1×1 mm^2 slits on both the incoming and the scattered beam. The measurements were performed in the middle of the specimens, in three orthogonal directions – one normal (z) and two in-plane (x and y) directions. Powder measurements were also performed in order to obtain the reference values.

The 2θ position of the Bragg peak is determined by a Gaussian fit. Using the Bragg's law $\lambda = 2d \sin\theta$ the strain can be calculated as:

$$\varepsilon = \frac{d - d_0}{d_0} = \frac{\sin\theta_0}{\sin\theta} - 1 \quad (1)$$

where d_0 is the unstrained interplanar distance of the considered lattice planes, while 2θ is the corresponding Bragg peak position which was determined by Gaussian fit of the data obtained from powder measurements.

Finally, the principal three orthogonal components of the residual stress were determined using Hooke's law:

Table 1
Densities and Young's modulus of the Cr(Re)/Al_2O_3 composites.

Grain size of Cr (μm)	Composite material	Measured density (g/cm^3)	Theoretical density (g/cm^3)	Relative density (%)	Young's modulus (GPa)
5	75%Cr + 25%Al_2O_3	6.31	6.38	99.0	280
	(75%Cr + 25%Al_2O_3) + 5%Re	7.04	7.11	99.2	310
40	75%Cr + 25%Al_2O_3	6.24	6.38	97.8	247
	(75%Cr + 25%Al_2O_3) + 5%Re	6.87	7.11	96.6	278