

Residual strain scanning of alumina-based ceramic composites by neutron diffraction

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Abstract. Residual strain profiles were measured by neutron diffraction in alumina-aluminium titanate ceramic composites sintered at two different temperatures, namely 1450 and 1550°C. The results show that irrespective of the direction and the sintering temperature, the obtained profiles are almost flat, with very similar results for both temperatures. In addition, the results demonstrate that the alumina is in compression whereas the aluminium titanate is subjected to tensile residual stresses.

1. Introduction

Ceramic composites have been investigated during decades to obtain improved mechanical performance. Some ceramic composites (Alumina Al_2O_3 - aluminium titanate Al_2TiO_5 composites) offer improved flaw tolerance and toughness [1-4], which is thought to be due to thermally-induced residual stresses. The average crystallographic thermal expansion of aluminium titanate is slightly higher than that of alumina ($\alpha_{\text{A}25-1000^\circ\text{C}} = 8.7 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$, $\alpha_{\text{AT}25-1000^\circ\text{C}} = 9.7 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$) and it is highly anisotropic. As a consequence, in an alumina-aluminium titanate composite, high tensile and/or compressive residual stresses are expected to develop among grains, depending on the particular relative orientations. These stresses might lead to a wide variety of possible toughening mechanisms: crack deflection, crack branching, crack bridging and microcracking. Therefore, the characteristics of the toughness curve and, as a consequence, the flaw tolerance of alumina-aluminium titanate composites may be strongly modified by appropriately altering the microstructure: grain size and aluminium titanate content.

Residual stresses have been measured by neutron diffraction in several ceramic systems [5-7]. However, residual stress profiles are seldom reported [7].

In this work, residual strain profiles were measured by neutron diffraction in 60 vol.% Al_2O_3 +40 vol.% Al_2TiO_5 ceramic composites (from now on A-AT40) prepared by slip casting and sintered at two different temperatures, namely 1450 °C and 1550 °C.



2. Experimental

2.1. Material

The investigated $\text{Al}_2\text{O}_3/\text{Al}_2\text{TiO}_5$ composites with 40 vol. % of Al_2TiO_5 were obtained using α - Al_2O_3 (Condea, HPA05, Houston, USA) and previously reacted Al_2TiO_5 powders fabricated using the same alumina and high purity anatase- TiO_2 (Merck, 808, Germany) powders. Plates with $70\text{mm} \times 70\text{mm} \times 10\text{mm}$ dimensions were obtained by slip casting, removed from the moulds and dried in air at room temperature for at least 24 h. In order to obtain the monolithic materials, the dried blocks were sintered in an electrical furnace (Termiber, Spain). The samples were heated at a rate of $2^\circ\text{C}/\text{min}$, with 4 hours dwell at 1200°C during heating and 3 hours dwell at the sintering temperature. Two different sintering temperatures, namely 1450°C and 1550°C , were used in order to obtain two different sets of samples. Sintered pieces were then machined to obtain test samples with final geometry of $30\text{mm} \times 30\text{mm} \times 10\text{mm}$.

2.2. Residual strain measurements

Residual strain measurements were performed by neutron diffraction on the strain imager SALSA (Strain Analyser for Large Scale engineering Applications) [8], at the ILL, Grenoble, France. SALSA features a Stewart platform as a sample stage, which allows very flexible sample handling capabilities and arbitrary scan trajectories in the 3D space. In our case, the (311) reflection of the double-focusing Si-monochromator was used at a take-off angle of 85° to obtain a wavelength of $\lambda = 2.06 \text{ \AA}$. A 2D position-sensitive detector with an angular opening of approximately 5° was mounted on SALSA. Primary and secondary slits were used to set the gauge volume (which can be varied automatically via step motors) of $1.5\text{mm} \times 1.5\text{mm} \times 10\text{mm}$. Strain scanning was carried out in both phases, Al_2O_3 and Al_2TiO_5 , in parallel (parallel to the largest surface of samples) and normal directions (perpendicular to the largest surface of the samples), with a scan step of 1 mm, as shown in Fig. 1.

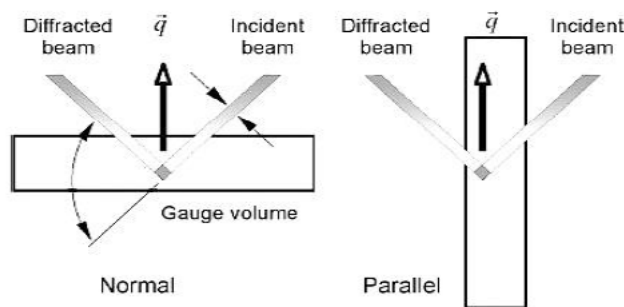


Fig. 1. Experimental setup employed in the neutron diffraction experiment for the normal and parallel directions

The (116) reflection was selected for Al_2O_3 , which corresponds to $2\theta=80.0^\circ$ for the above-mentioned wavelength. For Al_2TiO_5 , the (153) peak at $2\theta=87.9^\circ$ was selected, which is accompanied by a neighbouring peak (062) at $2\theta=87^\circ$. The LAMP software [9] was used to analyse the experimental results and obtain the peak parameters at each sample orientation.

According to Bragg's law, by precise measurements of the diffraction peak shift, the variation in the inter-planar spacing for a given $\{hkl\}$ set of planes can be evaluated and thus the elastic strain within the material can be calculated:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}} \quad (1)$$

where ε_{hkl} is the longitudinal strain in the direction of the scattering vector, and $d_{0,hkl}$ is the unstressed lattice spacing of the hkl reflection. The later was determined by measuring the single phase alumina

and aluminium titanate powders employed to fabricate the samples and the results are reported in Table 1.

Table 1. Unstressed lattice spacing values of the hkl reflections employed

	$\text{Al}_2\text{O}_3(116)$	$\text{Al}_2\text{TiO}_5(062)$	$\text{Al}_2\text{TiO}_5(153)$
$d_{0,hkl}$ (Å)	1.60365 ± 0.00014	1.4959 ± 0.0006	1.4840 ± 0.0003

3. Results and discussion

The residual strain profiles along the sample thickness are depicted in Fig. 2, for the two sintering temperatures, namely 1450°C (Fig. 2a) and 1550°C (Fig. 2b). The strain results for both sintering temperatures are very similar. This suggests that an increase of sintering temperature from 1450°C to 1550°C has no clear effect on residual strains for alumina-aluminium titanate monolithic composites.

Linear elasticity was employed to calculate residual stresses from residual strains [10]. The diffraction elastic constants of the alumina phase were calculated by a Kröner average for the (116) reflection ($E_{A,116} = 407$ GPa, $\nu_{A,116} = 0.21$), from the single crystal data published in the literature [11]. Since there are no reliable plane-specific diffraction elastic constants available for Al_2TiO_5 , bulk elastic constants ($E_{AT} = 155$ GPa, $\nu_{AT} = 0.33$), were used in the present work [12]. For the residual stress calculation, the (153) reflection of Al_2TiO_5 was chosen, because it was more intense and narrower than the (062) reflection.

Residual stresses profiles for the sample sintered at 1450°C are shown in Fig. 3. Stresses are not reported for the sample sintered at 1550°C because there are not enough measurements in the parallel direction. It can be seen that the stress profiles are almost flat, with tensile residual stresses in Al_2TiO_5 particles (average around 500 MPa) and compressive ones in the Al_2O_3 matrix (average around -160 MPa). The differences between normal and parallel stresses lie within experimental error in all cases. This suggests a hydrostatic residual stress state in both phases, which is consistent with the fabrication process.

Consequently, the calculated phase stresses should verify the equilibrium condition, i.e. negligible macro residual stresses [10]:

$$\sigma_M = f_A \sigma_A + f_{AT} \sigma_{AT} = 0 \quad (2)$$

where σ_M is the macro residual stress, f_A and f_T are the volume fractions of Al_2O_3 and Al_2TiO_5 , respectively, and σ_A and σ_{AT} are the corresponding phase stresses. From Fig. 3 it can be concluded that the calculated residual stresses approximately verify the equilibrium condition (Eq. 2).

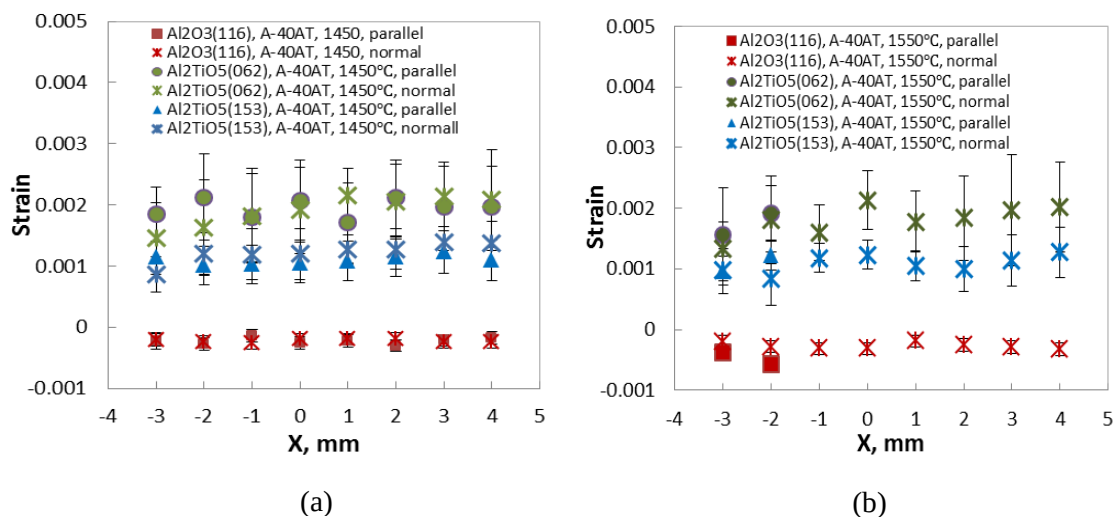


Figure 2. Residual strain (parallel and normal directions), (a) 1450 °C and (b) 1550 °C.

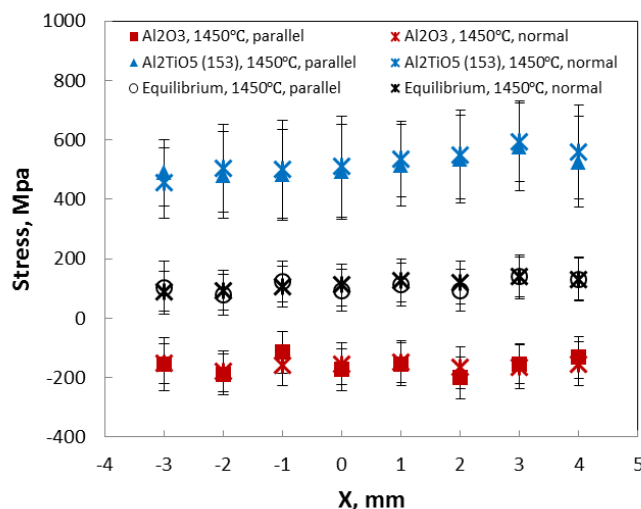


Figure 3. Residual stresses in parallel and normal directions for composites sintered at 1450 °C.

4. Conclusions

Residual strain scanning using neutron diffraction was used to calculate the residual stress profile in the alumina and aluminium titanate phases of $\text{Al}_2\text{O}_3 + 40 \text{ vol. } \%$ Al_2TiO_5 ceramic composites. The results show that alumina is under compression (around -160 MPa) and aluminum titanate is under tension (around 500 MPa). The residual stress state is hydrostatic in both phases. A change in the sintering temperature from 1450°C to 1550°C does not have a remarkable influence on residual strains. The calculated macro residual stresses are negligible for the sample sintered at 1450°C.

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