

# Toughening of the ceramic superconductor Bi-2223 with Ag

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## ABSTRACT

*In an attempt to increase the toughness of the high temperature superconductor  $\text{Bi}_{1.5}\text{Pb}_{0.5}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  (Bi-2223) we synthesised composites with three different filler morphologies of Ag. It is shown that Ag-addition improves Bi-2223 grain growth and reduces sample-porosity. Little effect on the mechanical strength is observed but the mismatch in thermal expansion coefficient induces a residual stress field at the filler matrix interface and allows toughening mechanisms to appear. This results in a clear post-peak behaviour when filler with high aspect ratio is used. The effect of the aspect ratio on the efficiency of the toughening mechanisms is studied.*

**KEYWORDS:** ceramic matrix composite, toughening, functional ceramic materials

## 1. INTRODUCTION

High temperature superconductors (HTSC) are brittle by their ceramic nature. This results in a possible cracking of the material through magnetostriction [Ikuta 1993].

It is well known that brittle ceramic material can be toughened by addition of a ductile material, if the precise balance in wetting and mismatch of thermal expansion coefficients between matrix and filler can be met. In the case of the reactive high temperature superconductors we have to consider an additional problem. These phases are only stable in a limited temperature range and are eager to react with secondary phases but non the less a high temperature treatment is required in order to avoid a negative effect on the electrical and magnetic properties. Earlier results obtained in composites of BSCCO with polyethylene and MgO [Bruneel 1998] were not satisfactory due to the loss of grain connectivity in the former and absence of toughening in the latter case. In this work Ag was chosen as filler for the superconducting  $\text{Bi}_{1.5}\text{Pb}_{0.5}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$  (Bi-2223) phase because it is one of the few metals that does not destroy the superconducting phase.

## 2. EXPERIMENTAL

### 2.1. Synthesis and experimental details

Ground superconducting 2223 powder [Van Driessche 1996] was mixed with either Ag-powder ( $< 50 \mu\text{m}$ ), Ag-wires ( $\text{Ø}: 0,05 \text{ mm}$ ) or Ag-whiskers in a 70:30 vol. % (Bi-2223:Ag) composition. The whiskers, with a diameter of 20-50  $\mu\text{m}$  and a length of a few hundred  $\mu\text{m}$ , were synthesised by an electrochemical reduction of an aqueous  $\text{AgNO}_3$  solution at pH 2 by a Cu wire (Fig. 1).

The mixtures were pressed into bar shapes of 2\*2\*13 mm with a pressure of 750 MPa and sintered for 3600 min. at 860°C. Three point bending tests were performed on an Instron™ series 4500 with a crosshead speed of 1 mm/min and a support span of 10 mm. SEM measurements were conducted on Philips 501equipment. The thermal expansion was measured with a TMA 2940 Thermomechanical Analyzer (TA Instruments).

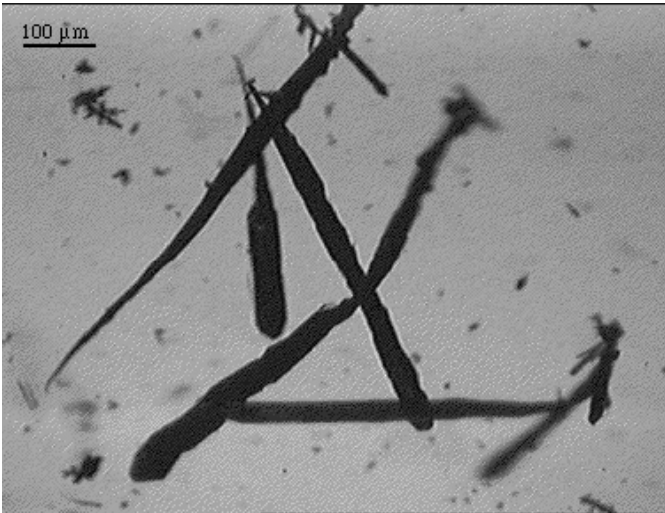


Figure 1. Ag-whiskers.

## 2.2. Results and discussion

From the mechanical measurements we can deduce that for all composites the modulus is approximately the weighted average of the monolithic phases (composite with 30 vol. % Ag grains:  $3600 \pm 1600$  MPa, density: 76 à 77,7 %), (Bi-2223:  $4800 \pm 2000$  MPa, density 69 à 75,6 %) and Ag (700 MPa, sintered Ag bar). This indicates that isostrain conditions are met, which is typical for a composite with a high modulus matrix and a low modulus filler [Schackelford 1998].

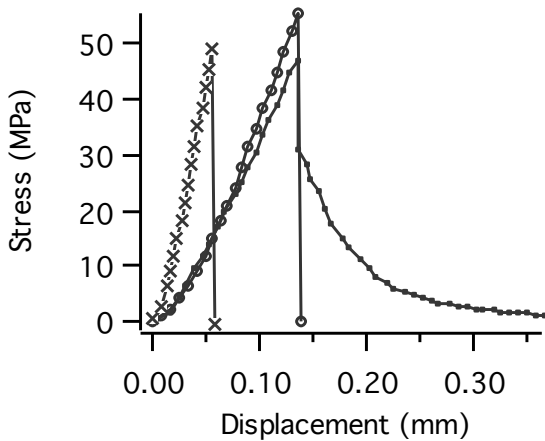


Figure 2. Representative stress -displacement curves of a monolithic (x) a particulate (O) and a whisker composite ( $\lambda$ ).

Table 1. Average of the total absorbed energy during 3-point bending tests.

	Absorbed energy (mJ)
Monolithic material	0,68
Composite with Ag grains	1
Composite with Ag whiskers	3,28
Composite with Ag wire	72

Addition of Ag-grains does not result in an appreciable post-peak behaviour in the stress/ displacement curve (Fig. 2), despite the observed crack deflection and crack bridging (Fig. 3). The particulate composites are tougher compared to the monolithic material thanks to the increased ultimate stress and the increase in strain to failure, table 1.

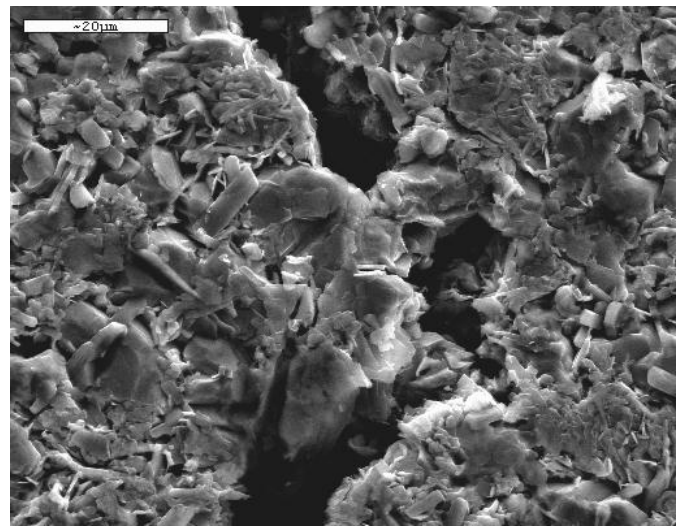


Figure 3. Crack bridging in composite with 30 vol. % Ag grains.

All Ag composites show increased 2223 grain growth as can be seen in Fig. 4. When used in moderate concentrations Ag has the ability to work as a sintering aid, this results in composites with a lower porosity than the monolithic material. This is considered to be the main reason for the slightly higher strength of the composites [Bruneel 2002]. The thermal expansion of the composite ( $13,3 \cdot 10^{-6} \text{ K}^{-1}$ ) is approximately the average of the thermal expansion of the constituent materials (Bi-2223:  $10,6 \cdot 10^{-6} \text{ K}^{-1}$  and Ag  $19,6 \cdot 10^{-6} \text{ K}^{-1}$ ).

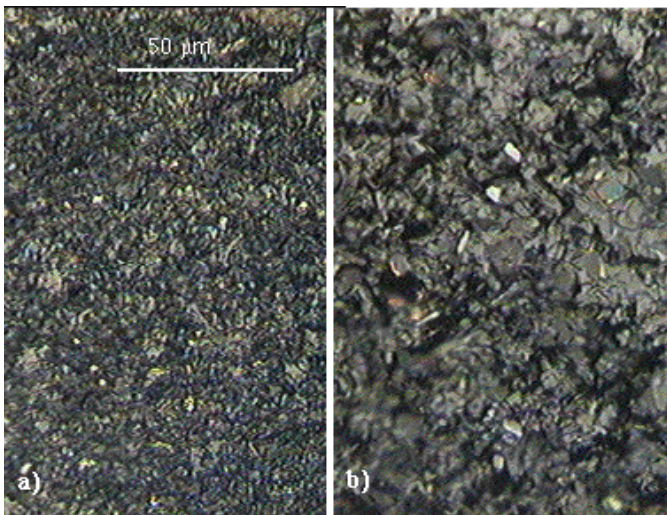


Figure 4: Surface of sintered bars a) monolithic Bi-2223, b) composite with 30 vol. % Ag grains. The marker has a length of 50  $\mu\text{m}$ .

In contrast with the measurements on composites with Ag-grains we observe a long tail in the stress-displacement curve of the whisker composites, indicating an increased toughness (Fig. 2). Due to whisker addition the area under the stress-strain curve is multiplied by 3. This difference in toughness between particle and whisker composites is attributed to the increase in aspect ratio, leading to more efficient toughening mechanisms. Fig. 5 shows crack bridging by Ag-whiskers. The higher aspect ratio of the wires makes the toughening mechanisms in the continuous unidirectional wire composites even more efficient than in the whisker composites. Fig. 6 shows the stress displacement curve of a wire-composite. The increase in absorbed energy and in displacement is now enormous. This is, again, ascribed to toughening mechanisms such as bridging and pull-out which can be observed in the SEM image in Fig. 7.

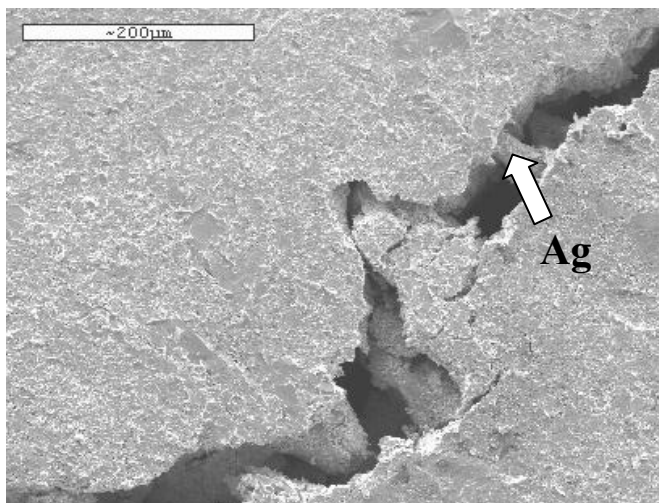


Figure 5. SEM, bridging by a Ag whisker.

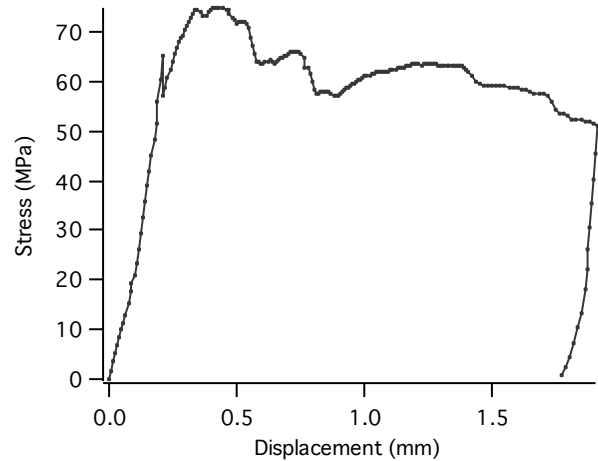


Figure 6. Representative stress displacement curve of continuous unidirectional composite.

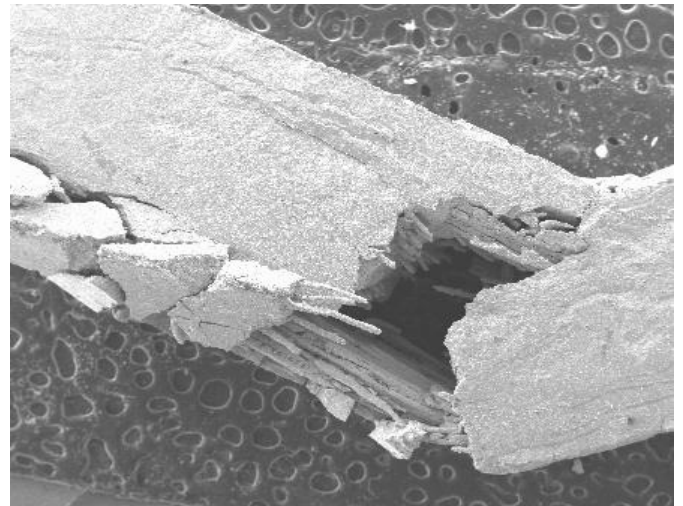


Figure 7. SEM, bridging and fibre pull out.

### 3. CONCLUSIONS

Particle, whisker and continuous unidirectional Ag wire composites based on the Bi-2223 superconducting phase were synthesised in order to make a tough superconducting material and to reveal the importance of the filler morphology. Three point bending tests show that isostrain conditions are met. In particulate composites the toughening is modest, but thanks to whisker and wire crack bridging and pull-out in whisker and wire composites these show an enormous enlargement of the absorbed energy, respectively multiplied by 3 and by 100 in contrast to monolithic material. These toughening mechanisms also result in an increased strain to failure.

We can conclude that toughening of the 2223 phase with high aspect ratio Ag was successful. The complex co-operation of the respective thermal expansion

coefficients, the properties of the interface, the morphology and the quantity of the filler leads to tougher, energy absorbing material.

#### 4. REFERENCES

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