Journal of Alloys and Compounds, 195 (1993) 679–682
JALCOM 7557

Toughness and thermoshock resistance of polycrystalline $YBa_2Cu_3O_{7-\delta}$

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1. Introduction

In non-cubic ceramics like YBaCuO, residual thermal mis-match stresses built up during cooling between neighbourhood grains and provok either stress-induced or spontaneous microcracking depending on the nature and the cohesive strength of the grain boundaries (1). Smith et al. (2) and Aslan et al. (3) have already made evidence of grain boundary cracking on the critical current density. These residual stresses influence the toughness which in turn and additionnally influences the thermal shock resistance. The laters will be measured here with the Vickers indentation method on various "microstructures" of polycrystalline YBaCuO. Furthermore since these residual stresses increase with decreasing temperature, the toughness will also be measured after quenching into liquid N_2 (77 K) in order to detect any irreversible change in their state, microcracking for example.

2. Materials

Mixtures of Y_2O_3, CuO and $BaCO_3$ have been calcinated at 880°C. After milling, the resulting powders were uniaxially pressed at room temperature (RT) and subsequently pressureless sintered at 920°C. Different microstructures have been obtained by using different soak times. The resulting discs (~ 5 mm thickness and 25 mm diameter) were prepared metallographically. The porosity was measured using Archimed's method in toluol.

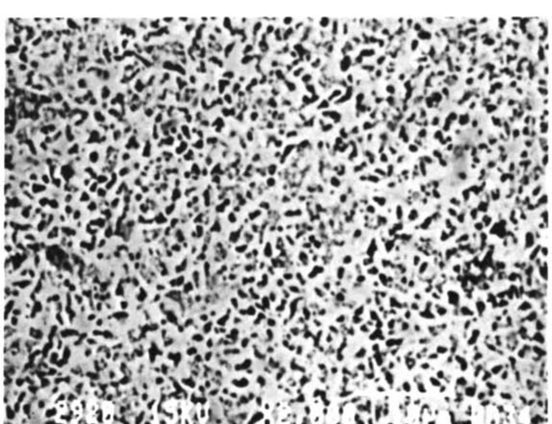
3. Results

3. 1. Toughness

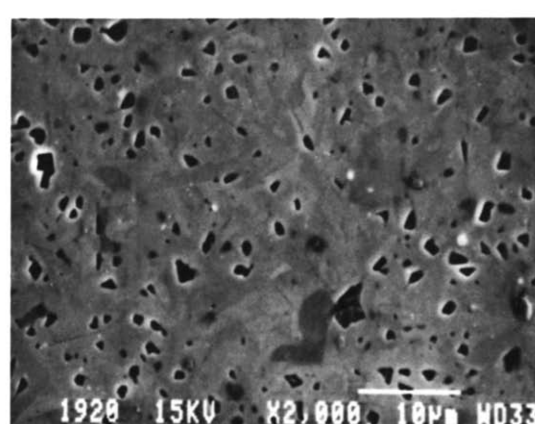
The importance of toughness has already been put in evidence (4) and measurements been made on single crystals with the microindentation method (5) or on polycrystals using the SENB specimen (6). In the present

Table 1 : Designation of the samples and main characteristics.

Sample	920 B	920 C	920 D	920 A
microstructure	very fine	fine	medium	coarse
porosity vol. %	19	19	22	16
Kc, MPa $\sqrt{m}$	0.70	0.84	1.64	0.24



Microstructure of sample 920 B



Microstructure of sample 920 A

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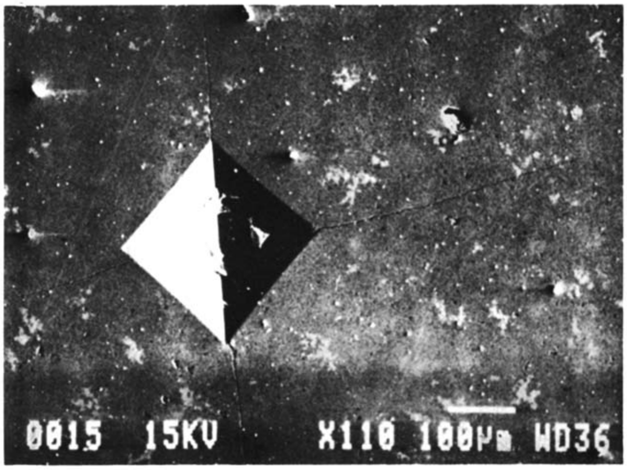

Fig. 1 : Vickers indentation on sample 920 B.

case the Vickers indentation method was used with loads ranging from 10 N to 100 N. After indentation a symmetric crack pattern appears (Fig. 1). A dimensional analysis (5) (7) yields a relation between indentation load, P, crack length, c, and the toughness of the material, Kc :

$$Kc = \xi(E/H)^{1/2}.P.c^{-3/2} = \chi r.P.c^{-3/2}$$

where E is Young's modulus and H, the hardness. ξ and χr are dimensionless proportionality factors. We used the microhardness value of Hv = 8.7 GPa (5) and E = 180 GPa (6) to verify wether the ratio (E/H) fits with the values used by Anstis et al., (7). The later obtained a ratio (E/H) ≈ 30 for a range of glassy and crystalline materials whereas (E/H) ≈ 20 holds in the present case. Thus toughness may be deduced from :

$$Kc = 0.77.P.c^{-3/2}$$

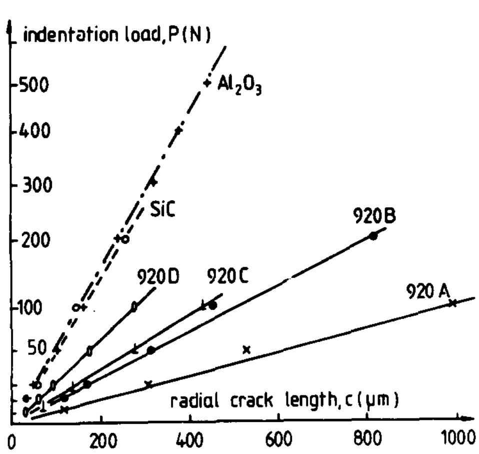

Fig. 2 : Relative toughnesses in $a_p^{2/3}$ vs. c plot.

In order to ensure the median-radial type of the cracks and to avoid discrepancies due to local inhomogeneities, the results, load and crack length, have been plotted in a $P^{2/3}$ vs. c diagram. The slopes of the straight lines obtained, proportional to $Kc^{2/3}$, is shown in figure 2 for the four microstructures, and compared to thermomechanical ceramics, Al_2O_3 (Kc = 3.8 MPa$\sqrt{m}$) and SIC (Kc = 3 MPa$\sqrt{m}$). The values of Kc are given in Table I.

3. 2. Thermal shock resistance

During rapid quenching of ceramic parts, transient tensile stresses develop on the surface (-or the skin-). Above a critical quenching temperature difference, ΔT_C, damage is introduced which provokes a decrease in strength. Such a procedure needs however a large number of samples with the same microstructure. This is not the case for materials in the R & D stage. Instead, we used a method consisting of measuring the increase in size of the radial cracks of the Vickers indentation. A detailed analysis of this procedure is given elsewhere (8). It is based on the fact that the radial cracks may extend in a stable way up to a given applied stress, decreasing with increasing indentation load and decreasing toughness of the material. Plots of the ratio (c/c_o) vs. indentation load, P, will thus yield graphs describing the relative resistances against a given thermal shock. Here c_o is the initial radial crack length and c its value after the thermal shock. We have quenched the indented samples from RT

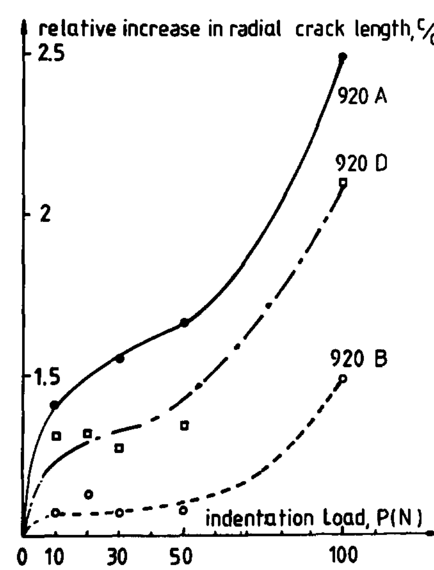

Fig. 3 : Relative resistances against thermal shock from RT into liq. N_2.

in air into liquid N_2 (77 K). The results are given figure 3 for three samples. Sample 920 A with the coarsest microstructure and the lowest value of toughness has the poorest resistance against quenching whereas sample 920 B which owns the finest microstructure and a higher value of toughness exhibits the smallest relative increase to approximatively the same transient thermal stress.

3.3. Toughness after quenching

After quenching, the toughness has been measured again with the same method outlined above. Results are shown in figure 4 for samples 920 A and 920 B after a first and second thermal shock into liquid N_2. Whereas the toughness of sample 920 B (finest microstructure) remains unchanged, does that of sample 920 A (coarsest microstructure) exhibit a sharp increase in slope, i.e. in toughness, after the first quenching. This indicates an irreversible change in the microstructure due to an increase of residual thermal mis-match stresses.

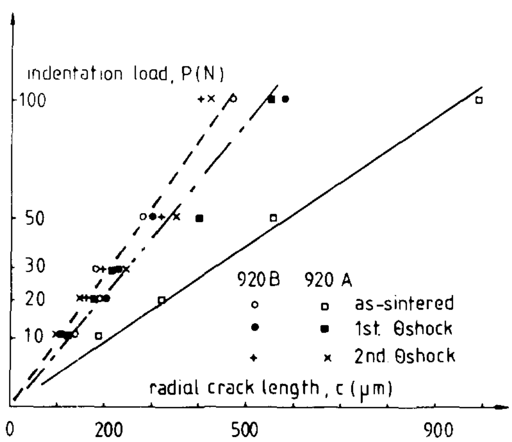

Fig. 4 : Detection of microstructural damage in coarse grained 920 A after cooling to liq. N_2.

4. Discussion and conclusion

The present work deals with the influence of microstructure on the toughness and thermal shock resistance of polycrystalline YBaCuO. The influence of grain size on the measured parameters can be correlated with the model of Rice and Freiman (9). This is based on the increased contribution of residual thermal mis-match stresses with increasing grain size :

- at small grain sizes, their contribution is small and toughness remains almost constant (920 B and 920 C)
- in a given range, their superimposition with the applied stress induces cracking of the grain boundary and an increase in toughness (920 D)
- above a critical grain size, their level is too high to need an appreciable level of applied stress for inducing microcracking and toughness thus decreases (920 A).

Thermal shock resistance may be related to toughness (see samples 920 B and 920 A). In figure 3, the ranking follows that of microstructure, indicating that in the case of 920 D, the increase in residual thermal mis-match stresses has lowered its toughness at 77 K (-see case of 920 A at RT).

Finally is the apparent increase in toughness of 920 A worth being discussed. The residual thermal mis-match stresses are already high at RT, so they may reach at 77 K a sufficient level for provoking spontaneous microcracking of the grain boundaries. First the highest are thus released and secondly the microcracks may pin or trap a main crack giving thus an indication of "high" toughness.

As a conclusion do the present results and method allow a throughout investigation of the thermomechanical behavior of polycrystalline YBaCuO, and other structural and functional, ceramics in the R & D stage.

Aknowledgment

This work is a part of the DAAD-PROCOPE founded project 311/Pro/CA.

1 D.R. Clarke, "Grain boundaries in polycrystalline ceramics", Ann. Rev. Mater. Sci. 17 (1987) 57.
2 D.S. Smith, S. Suasmore and C. Gault, "Demonstration of grain growth induced microcracking and its role in the electrical response of $YBa_2Cu_3O_{7-\delta}$", J. Eur. Ceram. Soc., 5 (1989), 81.
3 R. Aslan, H. Jarger, G. Kaiser, K. Gröner, K. Schulze and G. Petzow, "Influence of microstructure on the superconducting properties of polycrystalline $YBa_2Cu_3O_{7-\delta}$", J. Eur. Ceram. Soc., 6 (1990), 129.
4 D.R. Clarke, Th.M. Shaw and D. Dimos, "Issues in the processing of cuprate ceramic superconductors", J. Am. Ceram. Soc., 72 (7), (1989), 1103.
5 R.F. Cook, Th.R. Dinger and D.R. Clarke, "Fracture toughness measurements of $YBa_2Cu_3O_{7-\delta}$ single crystals", Appl. Phys. Lett., 51 (6), (1987), 454.
6 N. McN. Seford, J.D. Birchall, W.J. Clegg, M.A. Harmer, K. Kendall and S.H. Jones, "Physical and mechanical properties of $YBa_2Cu_3O_{7-\delta}$ superconductors", J. Mat. Sci., 23 (1988), 761.
7 G.A. Anstis, P. Chantikul, B.R. Lawn and D.B. Marshall, "A critical evaluation of indentation techniques for measuring fracture toughness : I, direct crach measurement", J. Am. Ceram. Soc., 64 (9), (1981), 533.
8 F. Osterstock, "Contact damage submitted to thermal shock : a mean to measure and simulate the thermal shock resistance of ceramics", This conference.
9 R.W. Rice and St.W. Freiman, "The grain size dependance of fracture energy in ceramics, II : a model for non-cubic materials", J. Am. Ceram. Soc., 64 (6), (1981), 350.