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DEFORMATION OF HIGH-TEMPERATURE SUPERCONDUCTORS

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INTRODUCTION

Of the many families of high-temperature superconductor, only the properties of those discovered prior to 1989 — Y-Ba-Cu-O, Tl-Ba(Sr)-Ca-Cu-O, and Bi(Pb)-Sr-Ca-Cu-O — have been studied extensively.¹ Deformation tests have been performed on YBa₂Cu₃O_x (Y-123), YBa₂Cu₄O_x (Y-124), TlBa₂Ca₂Cu₃O_x (Tl-1223), Bi₂Sr₂CuO_x (Bi-2201), Bi₂Sr₂CaCu₂O_x (Bi-2212), and (Bi,Pb)₂Sr₂Ca₂Cu₃O_x (Bi-2223).²⁻¹³ The tests have revealed that plasticity is generally limited in these compounds and that the rate-controlling diffusional kinetics for creep are very slow. Nevertheless, hot forming has proved to be quite successful for fabrication of bulk high-temperature superconductors, so long as deformation rates are low or large hydrostatic stresses are applied.¹³⁻²⁰

Steady-state creep data have proved to be useful in designing optimal heat treatments for superconductors and in support of more-fundamental diffusion experiments.^{21,22} The high-temperature superconductors are highly complex oxides, and it is a challenge to understand their deformation responses. In this paper, results of interest and operant creep mechanisms will be reviewed.

EXPERIMENTS AND MATERIALS

Most of the data cited are from our own work. Details of specimen preparation have been published.^{3,4,6,7,10-12} In brief, specimens were about 50% taller than wide and had an average grain size (*d*) of 5–70 μm. The Y-123 specimens were nearly fully dense and phase pure. The Tl-1223 specimens were 73–86% dense and ≈90% phase pure. The Bi-2201, 2212, and 2223 specimens were 92–99% dense; the Bi-2201 and 2212 were nearly phase pure and the 2223 was ≈90% pure.

The specimens were compressed between Al₂O₃ platens at nearly constant strain rate in an Instron machine fitted with a high-temperature furnace²³ or at constant load.^{4,6,12} In some cases, Ag or Pt foil was used as a diffusion barrier. For most specimens, more than

one steady state was established. Total strain was generally limited to $\approx 10\%$ and no appreciable specimen densification occurred during testing. Strain rates were based on initial sample length. Several deformed specimens were examined by scanning and transmission electron microscopy (SEM and TEM).

RESULTS AND DISCUSSION

All of the high-temperature superconductors have similar layered perovskite crystal structures (Fig. 1).¹ Cu-O planes, which are responsible for the superconductivity are common to all. As would be expected, creep properties of these compounds are generally similar. Results for Y-123 are by far the most extensive. These will be presented first and the results from the others will then be compared.

Creep of $\text{YBa}_2\text{Cu}_3\text{O}_x$

Y-123 is a highly oxidized compounds that is stable over only a rather limited range of temperature (T) and oxygen pressure (PO_2).²⁴ Reliable deformation tests have been performed from 750 to 980°C for specimens of various grain size d. The PO_2 range was 10^3 – 10^5 Pa.^{2-5,12,25,26} Steady state strain rate $\dot{\epsilon}$ was fit to a phenomenological expression

$$\dot{\epsilon} = A d^{-p} \sigma^n \text{PO}_2^m \exp(-Q/RT), \quad (1)$$

where A is a constant, p, n, and m are exponents, σ is the steady-state stress, Q is the activation energy, and R is the gas constant.²⁷

A range of values for the various parameters in Eq. 1 have been reported in the literature.^{2-4,9,12,26} For some time, the principal confusion, for which we were responsible, was the effect of temperature and PO_2 on Q. We reported initially that Q was a function of PO_2 .^{3,4} Recent experiments have resolved apparent discrepancies.¹² For Y-123, with $T > 900^\circ\text{C}$ and $\sigma < 10$ MPa,

$$\dot{\epsilon} = A d^{-2.7 \pm 0.6} \sigma^{1.0 \pm 0.1} \text{PO}_2^{-0.5 \pm 0.2} \exp(-1100 \pm 100 \text{ kJ/mole}/RT). \quad (2)$$

For $T \leq 880^\circ\text{C}$, the Q dropped to 675 ± 70 kJ/mole. Q depended only on temperature. The exponents n and m were independent of temperature. The value for p exhibited rather large scatter and has not been investigated thoroughly as a function of temperature. For Y-123 with submicron-sized grains, superplastic flow and an n value of 2.0 have been observed.^{8,9}

The measured creep parameters indicate a diffusional creep mechanism. No significant dislocation activity was observed in TEM and no change of grain shape was observed by SEM. Therefore, grain-boundary sliding (GBS) accommodated by diffusion was inferred to be the deformation mechanism. The Ashby-Verrall model for GBS in a compound can be expressed as

$$\dot{\epsilon} = A' (\sigma \Omega_{\text{mol}}/kT) d^{-2} D_{\text{mol}}, \quad (3)$$

where the threshold stress has been assumed to be negligible, $A' = 98$, Ω_{mol} is the molecular volume, and D_{mol} is the effective molecular diffusion coefficient, which takes into account diffusion in the bulk and along grain boundaries.^{12,28}

Equation 3 can be used to calculate diffusion coefficients directly.¹² Results of these calculations can be compared with measured tracer diffusivities.²² We take creep data from Ref. 4 at the highest temperatures, for which lattice diffusion should dominate, and lattice parameter data from Ref. 1: $d = 30 \mu\text{m}$, $T = 950^\circ\text{C}$, $\sigma = 1$ MPa, $\dot{\epsilon} = 10^{-6} \text{ s}^{-1}$, and

Figure 1. Crystal structures of $\text{YBa}_2\text{Cu}_3\text{O}_x$, $\text{TlBa}_2\text{Ca}_2\text{Cu}_3\text{O}_x$, and $\text{Bi}_2\text{Sr}_2\text{CuO}_x$, $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$, and $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$.

$\Omega_{\text{mol}} = 3.8 \times 3.9 \times 11.7 \text{ \AA}^3 = 2 \times 10^{-28} \text{ m}^3$. Thus, from data for high-temperature creep for constant PO_2 , we calculate the lattice diffusion coefficient (D_ℓ):

$$D_\ell = 7 \times 10^{35} \exp(-1100 \text{ kJ/mol/RT}) \text{ cm}^2/\text{s} . \quad (4)$$

Similar calculations can be made for grain-boundary diffusion,¹² but no grain-boundary diffusion data exist for Y-123. Equation 4 can be plotted against tracer diffusion data for Y-123 (Fig. 2).^{22,29,30} The agreement between diffusion coefficients calculated from creep data and measured diffusion coefficients for Y is excellent.

Two remaining questions are which defect is responsible for the rate-controlling diffusion — vacancies or interstitials — and the actual diffusion path. For ideal compounds in which simple point-defect theory holds, the PO_2 dependence can be used to deduce the diffusing species.³¹ Oxygen defects are by far the majority defect¹ in Y-123 and simple calculations would, therefore, ascribe the rate-controlling diffusion to Y interstitials.^{3,32} However, Y-123 is not a simple ionic compound, and it is doubtful that point-defect theory can be applied to each of the minority defects.²²

In a series of creep tests, 2% Ca^{+2} or Zr^{+4} was substituted for Y^{+3} in Y-123 and yet no consistent effect was observed on creep rate.²² Thus, charge compensation in the Y-123 crystal structure did not take place by creation of Y vacancies or interstitials. Although a large negative effect of increasing PO_2 on creep rate has been confirmed,^{3,12,22,26,32} the explanation for the effect remains unclear. Furthermore, examination of the crystal structure of Y-123 and comparison with other high-temperature superconductors¹ strongly suggests that Y vacancies are more likely to exist than Y interstitials.

Figure 2. Summary of tracer diffusion data for Y-123: diffusivities of ^{18}O , ^{64}Cu , and ^{133}Ba are shown as lines and are labeled; data points for ^{88}Y are shown as open squares; calculated diffusivity from creep data (D_ℓ) is shown as a dashed line; all data in O_2 .

Diffusion data reveal that the mechanism for diffusional creep in Y-123 is likely to be different from that of most ceramics.²⁷ Diffusion in Y-123 is many orders of magnitude faster in the a-b plane than in the c direction of the orthorhombic cell.²² In fact, diffusion of Y in the c direction is so slow that it has not yet been measured.²² Thus, during deformation, all of the diffusion must take place either through the bulk of the grains along a-b planes, or along grain boundaries (Fig. 3). This mixture of diffusion paths may account for the fact the grain size exponent obtained in creep is between 2 and 3.

Dislocation creep has also been observed for Y-123, but only under special conditions: Dislocation propagation within the basal a-b planes is rather easy, but is quite difficult in other directions.³³ Use of hydrostatic pressure or specially oriented, large-grained specimens can promote dislocation activity on non-basal planes.³⁴⁻³⁶ However, occurrence of general plasticity in Y-123 by dislocation motion has yet to be fully confirmed.²⁶ The dislocations introduced by deformation can, however, improve the flux pinning properties and, hence, critical current density.^{37,38}

Figure 3. Schematic diagram of diffusional flow in an isotropic ceramic and a high-temperature superconductor such as Y-123. The a-b planes in Y-123 are represented as parallel lines and diffusion within these planes is much faster than in any other direction.

Creep of $\text{YBa}_2\text{Cu}_3\text{O}_x$

Y-124 is structurally similar to Y-123. Its superconducting transition temperature is slightly lower, and a double Cu-O ribbon replaces a single Cu-O chainlike layer.³⁸ Creep tests have been performed on Y-124 in air and oxygen.¹³ Although apparent steady states were established, the stress exponent increased with decreasing temperature; from 880 to 950°C, n decreased from ≈ 1.9 to ≈ 1.1 . Although the average steady-state stress in Y-124 was about an order of magnitude higher than in Y-123 for specimens of approximately equal grain size, the estimated activation energy for diffusional creep was only ≈ 400 kJ/mole.¹³

No detailed microscopy has been conducted on deformed samples. Y-124 is not stable under the conditions of most of the creep tests, and decomposition to Y-123 + CuO would have been thermodynamically favored during most tests.^{39,40} The CuO decomposition products form in narrow platelets and other lattice defects are created by the decomposition.⁴⁰ It is possible that the low apparent activation energy for Y-124 is due to diffusion along short-circuiting paths created by these defects. In the absence of microstructural information, no firm conclusions on creep mechanism can be drawn.

Creep of $\text{TlBa}_2\text{Ca}_2\text{Cu}_3\text{O}_x$

The crystal structure of Tl-1223 is similar to that of Y-123 (Fig. 1). The principal differences are:^{1,41} (a) a Tl-O layer substitutes for a Cu-O chain layer, (b) instead of two square-planar Cu-O layers separated by Y atoms, one finds three square planar Cu-O layers, each being separated by Ca atoms, and (c) whereas Y-123 exhibits little range of cation substitution, there are appreciable solid-solution ranges in Tl-1223. These differences in structure are not profound, and one would expect the creep properties of Tl-1223 to be similar to those of Y-123.

The Tl-1223 samples that have been crept were rather porous and only ≈ 90 phase pure.⁷ In air, for strain rates $\leq 10^{-5} \text{ s}^{-1}$, stress exponents were 1.2 ± 0.15 for $T \geq 820^\circ\text{C}$ ($0.94 T_m$, where T_m is the peritectic melting point in air, $\approx 890^\circ\text{C}$). Stress exponents were higher at lower temperatures. TEM revealed substantial dislocation activity at temperatures $\leq 780^\circ\text{C}$ and some cracking. Little dislocation activity was observed at higher temperatures.

Deformation was attributed to diffusional flow at high temperatures and to a combination of dislocation motion and fracture at the lower temperatures. The creep data exhibited significant scatter attributable to poor specimen-to-specimen uniformity. Thus, the activation energy for diffusional flow could not be obtained accurately, but was on the order of 1 MJ/mol.⁷ Tl-1223 was somewhat more creep resistant than Y-123 (Fig. 4). For specimens of nearly identical grain size and equivalent homologous temperature, fully dense Y-123 crept slightly faster than $\approx 80\%$ dense Tl-1223.

Creep of $\text{Bi}_2\text{Sr}_2\text{CuO}_x$, $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$, and $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$

The crystal structure of Bi-2223 is very similar to that of Tl-1223. Sr substitutes for Ba and two Bi-O layers replace the single Tl-O layer (Fig. 1).¹ These Bi-O layers are weakly bonded, and all of the Bi cuprates cleave easily between the Bi-O layers. As a consequence, the grains of the Bi cuprates have much larger aspect ratios than are generally present in Y-123 or Tl cuprates (Fig. 5).

Creep studies have yielded two sets of results. For Bi-2212, with $\sigma < 3\text{--}4$ MPa and $T > 0.9 T_m$, diffusional flow has been observed.⁶ The activation energy was 990 ± 190 kJ/mol and the stress exponent was 1.0. No PO_2 dependence was observed for changes between 10^4 and 2.1×10^4 Pa O_2 . For equivalent $\dot{\epsilon}$, grain size, and homologous temperature, the steady-state σ of Bi-2212 was about an order of magnitude larger than was σ for Tl-1223.

Figure 4. Stress vs. strain rate for Tl-1223 creep tests: data from tests at equal temperature are denoted by identical symbol shape; individual specimens are denoted by manner in which each symbol is filled. 850°C corresponds to $0.95 T_m$ for Tl-1223; Y-123 data point for $0.95 T_m$ is shown as +.

Each of Bi-2201, 2212, and 2223 has reached apparent steady states for creep under higher stresses. Stress exponents were 3.1–3.8 and exhibited significant scatter.^{2,11} TEM and SEM revealed the presence of significant dislocation activity in crept specimens (Fig. 6), primarily in basal planes, and fracturing along basal planes. The fractures were both transgranular and intergranular. The data at higher stresses were sufficiently consistent that apparent activation energies could be calculated:¹¹

$$\begin{aligned}\text{Bi-2201} &= 520 \pm 50 \text{ kJ/mol,} \\ \text{Bi-2212} &= 630 \pm 210 \text{ kJ/mol,} \\ \text{Bi-2223} &= 960 \pm 210 \text{ kJ/mol.}\end{aligned}$$

The consistency of the data and TEM observations and the trend with temperature led to the conclusion that the kinetics of fracture of the specimens was controlled by dislocation motion within the basal planes. It appears as though dislocation motion is much more difficult in the Pb-doped Bi-2223 than in the Bi-2201 and 2212 compounds.

As with Y-123, it has been found that dislocations in Bi cuprates are effective in pinning flux and increasing critical current densities.⁴²⁻⁴⁴ These dislocations can be introduced at very low temperatures, but tend to anneal out with high-temperature heat treatment. The temperature range over which the dislocations are effective in pinning flux has been debated. All agree that dislocations are effective for $T < 40 \text{ K}$.⁴²⁻⁴⁴ For higher temperatures, dislocations may be relatively ineffective in pinning flux because of the two-dimensional nature of Bi-based cuprate superconductors: the dislocations would have to be present in every superconducting Cu-O layer, which is not possible.⁴⁵

Figure 5. TEM photomicrograph of Bi-2201 specimen consolidated by hot isostatic pressing.

Figure 6. TEM photomicrograph of dislocations at c-axis twist boundary in a crept Bi-2201 specimen; the parallel lines are Moiré fringes from interference between the two grains.

SUMMARY

Each of $\text{YBa}_2\text{Cu}_3\text{O}_x$, $\text{YBa}_2\text{Cu}_4\text{O}_x$, $\text{TlBa}_2\text{Cu}_2\text{Cu}_3\text{O}_x$, $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$, $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$, and $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ has been shown to deform at $T > 0.9 T_c$ by diffusional flow. For all but $\text{YBa}_2\text{Cu}_4\text{O}_x$, the activation energy for creep was $\sim 1 \text{ MJ/mole}$. For $\text{YBa}_2\text{Cu}_3\text{O}_x$, creep rates increased with decreasing P_{O_2} ; few data exist for other samples. Most of the other high-temperature superconductors

All of these compounds have complex layered perovskite crystal structures. Diffusion appears to take place through the bulk of the grains, almost exclusively within the a-b planes, and along grain boundaries. Dislocations move relatively easily within the basal planes, but not in other directions. Generalized plasticity through dislocation motion is very difficult to accomplish in the absence of large hydrostatic pressures. Nevertheless, plastic deformation is a useful means of fabrication and for improving properties of high-temperature superconductors.

Recommendation for future work on deformation of high-temperature superconductors include: systematic investigation of PO_2 effects in superconductors other than Y-123; tensile testing of polycrystals; compression testing of large single crystals, and testing those compounds not yet deformed.

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