

NASA Technical Memorandum 101401

# Stability of Bulk $Ba_2YCu_3O_{7-x}$ in a Variety of Environments

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Prepared for the  
Fall Meeting of the Materials Research Society  
Boston, Massachusetts, November 28—December 2, 1988

N89-14310

Unclass  
0174994

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(NASA-TM-101401) STABILITY OF BULK  
 $Ba_2YCu_3O_{7-x}$  IN A VARIETY OF ENVIRONMENTS  
(NASA) 13 F CSCI 11C



## STABILITY OF BULK $Ba_2YCu_3O_{7-x}$ IN A VARIETY OF ENVIRONMENTS

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### SUMMARY

Small bars (1 by 1 by 14 mm) of ceramic  $Ba_2YCu_3O_{7-x}$  were fabricated and subjected to environments similar to those that might be encountered during some NASA missions. These conditions include ambient conditions, high humidity (100 percent humidity up to 50 °C), vacuum ( $10^{-4}$  Pa), and high fluences of electrons ( $9.7 \times 10^{14}$  e<sup>-</sup>/cm<sup>2</sup>) and protons ( $8.7 \times 10^{14}$  p<sup>+</sup>/cm<sup>2</sup>). The normal state resistivity or critical current density ( $J_C$ ) were monitored during these tests to assess the stability of the material under these conditions.

When normal state resistivity is used as a criterion, the ambient stability of these samples was relatively good, exhibiting only a 2 percent degradation over a 3 month period. The humidity stability was shown to be very poor, and to be a steep function of temperature. Samples stored at 50 °C for 40 min increased in normal state resistivity by four orders of magnitude. Kinetic analysis indicates that the degradation reaction is second order with water vapor concentration. It is suspected that humidity degradation also accounts for the ambient instability. The samples were stable to vacuum over a period of at least 3 months.

Degradation of  $J_C$  (defined as the current density that produced a potential greater than 1  $\mu$ V/mm) in an 1 MeV electron fluence of  $9.7 \times 10^{14}$  e<sup>-</sup>/cm<sup>2</sup> was determined to be no more than about 2 percent. Degradation of  $J_C$  in a  $8.7 \times 10^{14}$  p<sup>+</sup>/cm<sup>2</sup> of 42 MeV protons was found to be grain size dependent. Samples with smaller grain size and initial  $J_C$  (77 K) of about 240 A/cm<sup>2</sup> showed no degradation, while that with larger grain size and an initial  $J_C$  (77 K) of about 30 A/cm<sup>2</sup> degraded to 37 percent of its original value.

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\*Work supported by NASA Grant NCC-3-19.

## INTRODUCTION

It is commonly believed that space technology will be one of the first arenas where the perovskite-type high temperature superconductors will see their applications. This is because component costs are not as critical as performance, since their costs are likely to be a small fraction of the mission, while mission success may hinge on their performance.

Materials which are used in spacecraft must be able to withstand a wide variety of conditions. These include typical earth environmental factors, such as atmospheric oxygen and water, which any material must either be subjected to or protected from during manufacture and assembly. Launch conditions add to these factors vibrational stresses and high g-force structural loads. If the payload is in a low earth orbit, there is moderately high vacuum ( $10^{-5}$  Pa) and the residual atmosphere is dominated by a very corrosive gas, atomic oxygen. At high earth orbits and deep space there is high vacuum ( $10^{-8}$  Pa), and radiation fluxes from the solar wind. The radiation fluxes are particularly high in the radiation belts which surround the earth and other planets. If the spacecraft is a planetary probe, it must be able to withstand temperatures, pressures and atmospheric constituents of the target planet. These range from the hot oxidizing environment of Venus, to the cold reducing environments of Jupiter and the other gas giants.

If the intent is to utilize high temperature superconductors on spacecraft, it must first be determined how vulnerable they are to this variety of conditions. If the materials are vulnerable, additional measures which are required to protect the material may be determining factors as to whether an application is feasible. This study is an initial attempt to assess the vulnerability of one of the high temperature superconducting materials,  $\text{Ba}_2\text{YCu}_3\text{O}_{7-x}$ , to some of these conditions.

There were several limitations to this study which should be recognized from the outset. The first is that the materials investigated were experimental. There are relatively large batch to batch variations and probably some minor phases are present (below the limit of detection using x-ray diffraction). When a standard material becomes available it will no doubt be of higher quality. Since this study used ceramic materials, and not single crystals, some of the observed effects may be artifacts of grain boundaries which will change as the material quality goes up. Nevertheless, it seems reasonable to begin to screen these materials at this early stage to point out possible trouble areas, and areas in which even these crude materials perform well.

## METHODS AND MATERIALS

Powders of  $\text{Ba}_2\text{YCu}_3\text{O}_{7-x}$  were synthesized with  $\text{BaO}_2$ ,  $\text{Y}_2\text{O}_3$ , and  $\text{CuO}$  using standard techniques (ref. 1). For comparison, powders were also provided by Ferro Corp. The powders were pressed into bars measuring 1 by 1 by 14 mm at room temperature using a pressure of 200 to 350 MPa. The bars were sintered at 900 °C for 16 hr, and then annealed at 450 °C in an oxygen flow for 8 hr. Samples were stored in a desiccator until used.

Two types of contacts were used, depending on the sample properties to be measured. For the samples which only had  $T_c$  and normal state resistivity

measured, silver paint was used to paint contacts on the bars after they were annealed. For those which had  $J_c$  measured, silver epoxy contacts were applied after sintering, and were then annealed on.

The resistivity of the samples was measured using conventional four point techniques with a Keithley Model 220 constant current source and a Keithley Model 181 nanovoltmeter. The voltage was measured at both polarities to negate electrochemical effects.

Even though normal state resistivity itself is not a technologically useful parameter with these materials, we have found that it is indicative of the superconducting properties. Samples with deteriorating normal state properties will also have deteriorating superconducting properties. Moreover, normal state resistivity can be measured in-situ, and does not subject the sample to temperature cycling, which we suspect may degrade the sample. We believe the normal state resistivity to be a fairly sensitive indicator of the condition of the sample, since it is measured from the surface where the effects of degradation would most likely be seen first. However, since these samples have much smaller surface to volume ratios than would be encountered in thin films, thin film degradation would no doubt occur much faster.

The transport critical current density ( $J_c$ ) was measured at 77 K with the sample submerged in liquid nitrogen using a Keithley Model 228 constant current source, and a Keithley Model 181 nanovoltmeter under computer control. Current-voltage (I-V) curves were generated by stepping the current in increments of 0.01 A from the normal state of one polarity through the superconducting transition and back to the normal state at the other polarity. Sample holders were designed such that the sample could be subjected to the environmental condition and then measured repeatedly without having to remount the sample.

The authors believe that transport  $J_c$  at 77 K is the critical parameter to be monitored. Since devices will probably be used at this temperature and since  $J_c$  is the limiting parameter for these applications, this measurement is the most stringent.  $J_c$  was defined in these experiments as the current density that produced a potential greater than 1  $\mu\text{V}/\text{mm}$  of length.  $J_c$  was also found to be much more sensitive to the effects of degradation than critical temperature ( $T_c$ ). This is because a sample will show zero resistance at  $T_c$  if there is any connecting conduction path between the voltage probes, but  $J_c$  is dependent upon the amount of material which is superconducting. Transport  $J_c$  is not only the parameter that is required for engineering specifications, but it gives insights into the grain boundary interactions. Grain boundary interactions dominate the superconducting properties of ceramic materials.

After it was confirmed that the samples were superconducting, and had  $T_c$  in excess of 90 K, they were mounted on four point resistivity sample boards (fig. 1). The samples were then left out in the ambient air and there normal state resistivity was measured at increasing time intervals.

Additional samples were mounted on sample boards which were attached to vacuum feed-throughs. The feed-throughs were mounted to a vacuum line and evacuated with a turbomolecular pump (TMP) to a pressure of about  $10^{-5}$  Pa. This is approximately the vacuum of low earth orbit. The four point resistivity of the samples could be measured without removing them from vacuum. After

17 days there was a failure of the TMP. So data beyond this time represents exposure to a roughing vacuum of about  $10^{-3}$  Pa.

Other samples were mounted on sample boards and placed in humidity chambers (fig. 2). These consist of two beakers, one with the other. The smaller beaker was partially filled with water, and the sample sat on top of it. This was placed with a larger beaker with the electrical leads trailing out and the large beaker was sealed with Parafilm. This assembly was in turn placed within an incubator set at the test temperature. The chambers were allowed to equilibrate overnight at the test temperature before the samples were added. A chart recorder was connected to the nanovoltmeter because of the speed of the reaction.

Electron irradiations were carried out at the Westinghouse Research Center in Pittsburgh with the Van de Graaff generator. Electrons of 1 MeV energy were used and the flux was measured using standard Faraday cup techniques. Control samples were exposed to the same environment as the test samples, less the radiation. Samples were irradiated to a total fluence of  $9.7 \times 10^{14}$  e<sup>-</sup>/cm<sup>2</sup> in three irradiations over a one month period.  $J_c$ 's of the samples were measured after fluences of  $7 \times 10^{13}$  and  $2.7 \times 10^{14}$  e<sup>-</sup>/cm<sup>2</sup> as well as at the total fluence level.

Proton irradiations were carried out at the NASA Lewis Research Center cyclotron, which is operated by the Cleveland Clinic Foundation. Samples were mounted on a test fixture (fig. 3) which allowed them to be irradiated, and then submerged immediately into a dewar of liquid nitrogen. Desiccant was placed at the bottom of the dewar so that when the nitrogen diffused out of it, the samples remained dry.  $J_c$  was measured on the samples without disturbing them on the test fixture.

Both the electron and proton fluences tested here are equivalent to what the material might see in the hottest portions of the Van Allen belts during a period of maximum solar activity for about a year. Typical radiation levels in low earth orbit may be as many as seven orders of magnitude lower.

## RESULTS AND DISCUSSION

### Ambient

It has been apparent from the earliest work that these materials are sensitive to the ambient environment. Kitazawa et al. have discussed the problem in some detail (ref. 2). They find there is an enhancement of the degradation when both water and carbon dioxide are in the environment. They did not, however, quantify this deterioration for samples which were simply exposed to the ambient laboratory air. It was important for us to determine the effect of the ambient air on our samples both as an internal control measure, and also as a guide for other researchers.

As expected, degradation of the normal state resistivity when the samples were stored at room temperature was observed. As is illustrated in Fig. 4, the resistivity increases by about 4 percent over a seven week period. The amount of degradation is no doubt dependent on the surface to volume ratio.

In these samples the surface to volume ratio was about  $40 \text{ cm}^2/\text{cm}^3$ . For films and fibers this number would be much higher, and degradation would be expected to be faster.

We also monitored the resistivity of samples stored in a desiccator with a  $\text{CaSO}_4$  drying agent. Although stability was improved, Fig. 4 shows that there was still a normal state resistivity increase of nearly 2 percent over this same time period.

#### Vacuum

Although it has been apparent that the oxygen in  $\text{Ba}_2\text{YCu}_3\text{O}_{7-x}$  is labile at high temperatures, little is known about the behavior of the material in vacuum for long time periods at room temperature and below. Kroeger et al., however, have observed compositional and morphological changes on fracture surfaces with time in vacuum at room temperature using Auger electron spectroscopy even in the absence of electron beam excitation (ref. 3).

The tests in this study show very little, if any, degradation in the normal state resistivity of the samples on exposure to vacuum (fig. 4). The changes reported by Kroeger et al. occur very near the surface and have negligible effect on the bulk, at least over time spans of a few weeks. However, the vacuum under which our tests were conducted was not as great as that in the other study. It is still possible that, in deep space vacuum after several years, effects could surface. These would most seriously effect thin films, with their high surface to volume ratios.

#### Humidity

There have been many reports in the literature about the instability of  $\text{Ba}_2\text{YCu}_3\text{O}_{7-x}$  to water. Interestingly, it has been reported to be inert to submersion in cold water for several hours (ref. 4), though even slight heating ( $35^\circ\text{C}$ ) causes a rapid decomposition into  $\text{BaY}_2\text{CuO}_5$ ,  $\text{Ba}(\text{OH})_2$ , and  $\text{O}_2$ . The  $\text{Ba}(\text{OH})_2$  then reacts with  $\text{CO}_2$  to form  $\text{BaCO}_3$  (ref. 5).

A more troubling problem than immersion is the reaction with water vapor in the air. Dexin et al. reported that exposure of a sample in air at 100 percent humidity at  $60^\circ\text{C}$  for 20 min resulted in needle crystals growing from crystal grain edges (ref. 6). We saw the same reaction. They further report that  $T_c$  (zero) drops with time (to 50 K after 80 min), but that  $T_c$  (onset) is nearly unaffected.

This study was similar to that of Dexin et al., except that the temperature of the reaction was also varied. The results are summarized in figure 5, which shows the log of the resistivity/initial resistivity (assuming no dimensional changes) as a function of time at 30, 40, and  $50^\circ\text{C}$ , 100 percent humidity.

If normal state conductivity is used as an indicator of the extent of reaction, interesting trends emerge. When raising the temperature in these systems there are two effects; raising the average kinetic energy of the molecules, and increasing the vapor pressure of the water. A change from 30 to  $50^\circ\text{C}$  changes the kinetic energy of the gas phase molecules by less than

7 percent, but increases the vapor pressure by a factor of three. If, therefore, increasing the temperature is taken to have only the effect of changing the available concentration of water molecules, the data are found to lie along a straight line on a graph of vapor pressure of water as a function of the reciprocal of the time to reduce the conductivity by half (fig. 6). This implies a second order reaction.

This surprising result can lead to many different mechanisms. We speculate that perhaps the species which initiates the decomposition is not  $H_2O$ , but actually the  $H_3O^+ - OH$  couple, with the  $OH^-$  abstracting the Ba, and the  $H_3O^+$  acting to stabilize the activated transition complex. The fact that the compounds show little decomposition when exposed to NaOH solutions suggests that the  $H_3O^+$  is important (ref. 7).

Perhaps a second order mechanism simply reflects the divalency of the barium ion, which must capture two water hydroxide ions to be debonded from the  $Ba_2YCu_3O_{7-x}$  lattice. Without more data, there are few constraints on other scenarios.

### Electron Irradiation

There have been at least two studies of the effects of electron irradiation on  $Ba_2YCu_3O_{7-x}$ . Maisch et al. irradiated thick films with very high energy electrons (55.7 MeV), and found at high fluences (100 mrad) that the room temperature resistivity increased by a factor of 2 to 3, and became more semiconductor-like in its temperature dependence. They observed no shift in  $T_C$  (onset), but a decrease of about 5 K in  $T_C$  (zero) (ref. 8).

Recently, Adem et al. reported a study using 1 MeV electrons at fluences of  $6.5 \times 10^{11}$  and  $1.7 \times 10^{15}$  e-/cm<sup>2</sup> (ref. 9). They also reported a decrease of  $T_C$  (zero) to 52 and 40 K respectively, no substantial change in  $T_C$  (onset), and that the samples behaved more semi-conductor-like. They further reported increasing disorder in the lattice and rounding of grain boundaries caused by the irradiation.

In this study we looked at  $J_C$  as a function of irradiation with 1 MeV electrons (fig. 7). It was found that fluences up to  $9.7 \times 10^{14}$  e-/cm<sup>2</sup> degraded  $J_C$  (77 K) by no more than about 2 percent over the control samples. A limited number of samples (six) were used, so even this degradation could be within the deviation of the samples. The control samples showed some degradation also, probably due to exposure to the air during the irradiations.

### Proton Irradiation

The effects of proton irradiation at a variety of proton energies has been studied in a preliminary way. Willis et al. studied sintered pellets of  $Ba_2GdCu_3O_{7-x}$  and  $Ba_2EuCu_3O_{7-x}$  irradiated with 800 MeV protons at 90 °C (ref. 10).  $T_C$  (onset) for both compounds dropped at a rate of (1.7 K)/ $10^{17}$  p+/cm<sup>2</sup>. SQUID measurements showed that flux expulsion decreased from 20 percent to less than 10 percent after  $10^{17}$  p+/cm<sup>2</sup>. Intragrain  $J_C$  was found to increase by a factor of three in zero field.

In another study, Maisch et al., studied thick films (100  $\mu\text{m}$ ) of  $\text{Ba}_2\text{YCu}_3\text{O}_{7-x}$  irradiated with protons of 63 MeV up to a fluence of  $1.09 \times 10^{14}$   $\text{p}^+/\text{cm}^2$ , and with 6 MeV protons to a fluence of  $5.0 \times 10^{13}$   $\text{p}^+/\text{cm}^2$  (ref. 6). With the 63 MeV samples it was found that at fluences as low as  $7.8 \times 10^{12}$   $\text{p}^+/\text{cm}^2$ , there was no  $T_c$  (zero) above 4.2 K. Except for a small random disordering of the lattice, there was no change on the structure as detected by x-ray diffraction. It was difficult to interpret the 6 MeV proton data because the penetration depth (150  $\mu\text{m}$ ) was similar to the sample thickness.

In this study, 42 MeV proton irradiation revealed a stability effect that was not observed in any of the other tests. There were two sample types for these tests. Samples produced at NASA had large grains and lower initial  $J_c$  values ( $\approx 30$   $\text{A}/\text{cm}^2$ ). Samples produced at Ferro had small grains and higher initial  $J_c$  values ( $\approx 240$   $\text{A}/\text{cm}^2$ ). The 42 MeV protons proved much more damaging to the large grain samples (fig. 8). After a fluence of  $8.7 \times 10^{14}$   $\text{p}^+/\text{cm}^2$  the small grain samples showed essentially no degradation, whereas the large grain samples degraded to about one-third of their original  $J_c$ . We think that the larger grain size makes the NASA produced samples much more susceptible to proton radiation than the Ferro produced samples.

## CONCLUSIONS

There are some practical handling techniques suggested by this study. First, this study confirms the results of others that atmospheric water vapor should be avoided. Although the material does not degrade so rapidly as to prohibit experimentation outside of a glove box (at least at this surface to volume ratio), it does suggest that long term storage in vacuum is superior to storing them in a desiccator.

The irradiation experiments indicate that, except for the large grained samples, the materials are reasonably hard to the radiation they are likely to see in space. What their lifetime can be expected to be is difficult to determine without detailed knowledge of the specific mission. Additional studies are required to determine the failure points for both proton and electron radiation, and to verify if the grain size effect is real.

## ACKNOWLEDGEMENTS

The authors would like to acknowledge the kind contribution of powders of  $\text{Ba}_2\text{YCu}_3\text{O}_{7-x}$  by M.A. Centanni of the Ferro Corporation Technical Center, Independence, Ohio. One of us (PDH) would also like to acknowledge the support of NASA under Grant # NCC-3-19.

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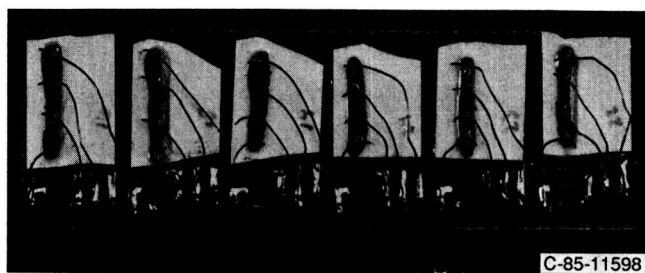


Figure 1. - Sample holder for making four-point resistivity measurements on ceramic bars of  $Ba_2YCu_3O_{7-x}$ .

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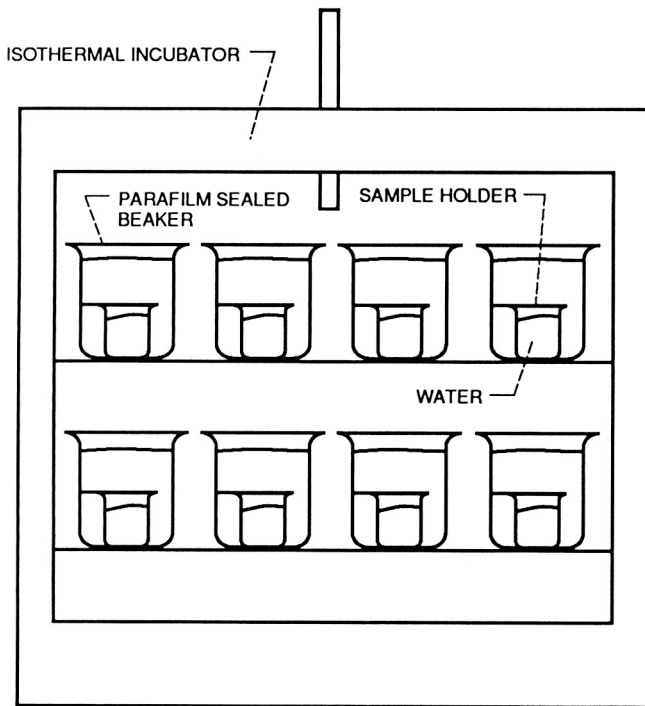


Figure 2. - Humidity chambers used for testing the stability of ceramic bars of  $Ba_2YCu_3O_{7-x}$ .

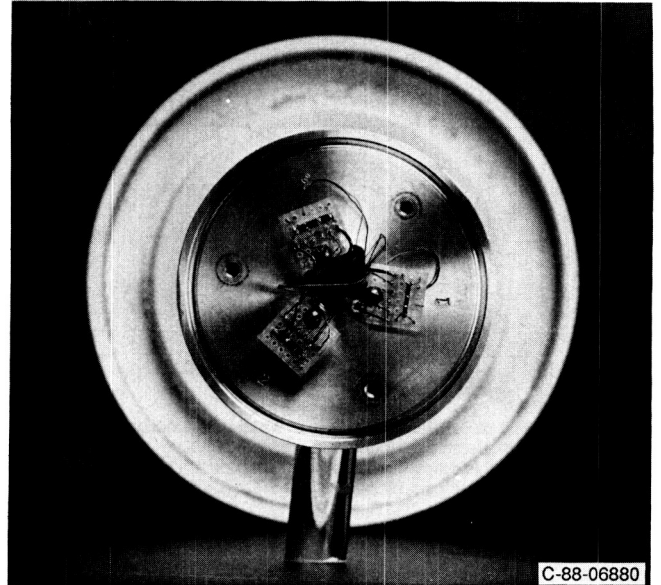


Figure 3. - Test fixture to irradiate ceramic bars  $Ba_2YCu_3O_{7-x}$  in a cyclotron, and then measure their critical current density.

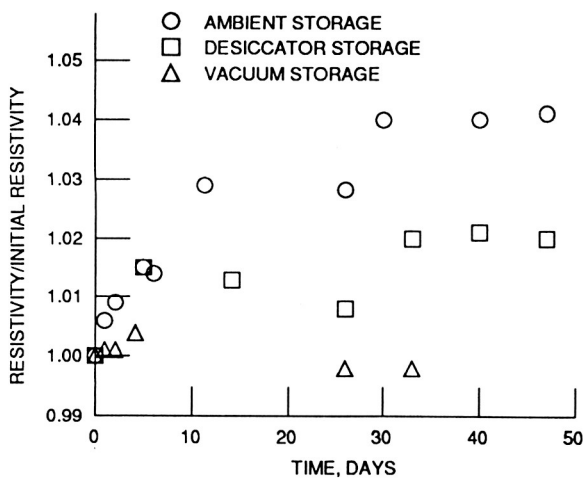


Figure 4. - Degradation of ceramic bars of  $Ba_2YCu_3O_{7-x}$  on exposure to ambient conditions, desiccated air, or vacuum over time.

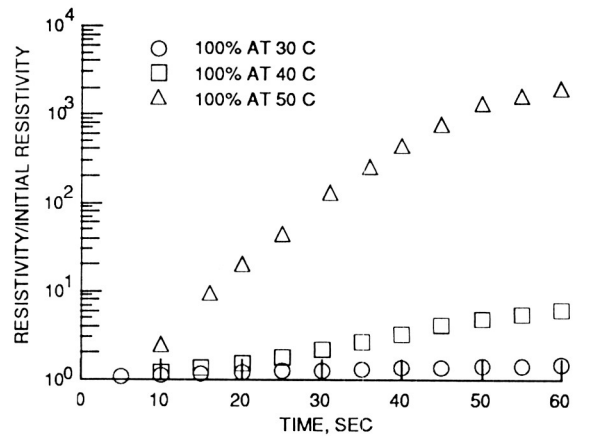


Figure 5. - Degradation of ceramic bars of  $Ba_2YCu_3O_{7-x}$  on exposure to 100 percent humidified air, at 30, 40, and 50 °C over time.

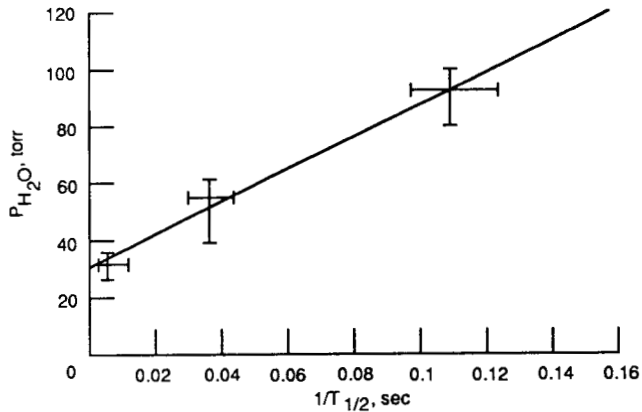


Figure 6. - Partial pressure of water as a function of the reciprocal of the time to degrade the sample conductivity by half. Linearity in this graph indicates a second order chemical reaction of water with the ceramic bars of  $Ba_2YCu_3O_{7-x}$ .

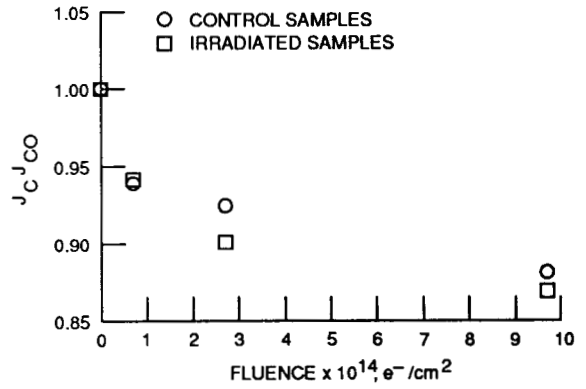


Figure 7. - Degradation of ceramic bars of  $Ba_2YCu_3O_{7-x}$  on exposure to 1 MeV electron irradiation.

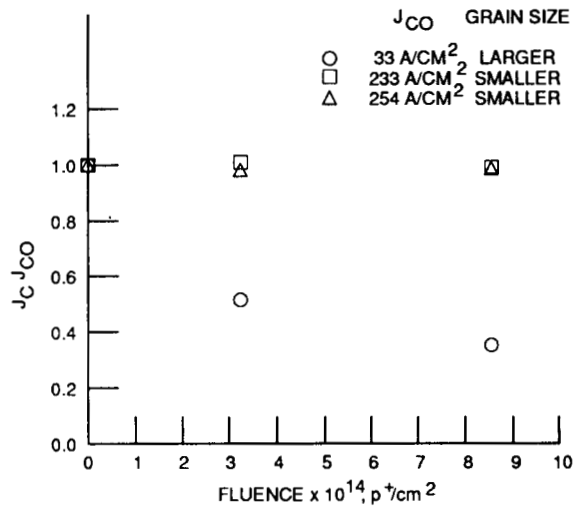


Figure 8. - Degradation of ceramic bars of  $Ba_2YCu_3O_{7-x}$  on exposure to 42 MeV proton irradiation.

1. Report No. NASA TM-101401		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle Stability of Bulk Ba <sub>2</sub> YCu <sub>3</sub> O <sub>7-x</sub> in a Variety of Environments				5. Report Date	
				6. Performing Organization Code	
7. Author(s) James R. Gaier, Aloysius F. Hepp, Henry B. Curtis, Donald A. Schupp, Paul D. Hamburger, and James W. Blue				8. Performing Organization Report No. E-4485	
				10. Work Unit No. 506-41-41	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546-0001				14. Sponsoring Agency Code	
15. Supplementary Notes  Prepared for the Fall Meeting of the Materials Research Society, Boston, Massachusetts, November 28—December 2, 1988. James R. Gaier, Aloysius F. Hepp, and Henry B. Curtis, NASA Lewis Research Center; Donald A. Schupp, Rensselaer Polytechnic Institute, Troy, New York 12180; Paul D. Hamburger, Cleveland State University, Cleveland, Ohio 44115 (work supported by NASA Grant NCC-3-19); James W. Blue, Cleveland Clinic Foundation, Cleveland, Ohio 44106.					
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17. Key Words (Suggested by Author(s)) High temperature superconductors Stability Radiation hardness			18. Distribution Statement Unclassified—Unlimited Subject Category 27		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No of pages 12	22. Price* A03