MTL TR 90-51

AD

HOT ISOSTATIC PRESSING OF SUPERCONDUCTING CERAMICS

KERRY T. RICHARDS and CPT RICHARD H. BENFER
CERAMICS RESEARCH BRANCH

October 1990

US ARMY
LABORATORY COMMAND
MATERIALS TECHNOLOGY LABORATORY

U.S. ARMY MATERIALS TECHNOLOGY LABORATORY
Watertown, Massachusetts 02172-0001

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

Mention of any trade names or manufacturers in this report shall not be construed as advertising nor as an official indorsement or approval of such products or companies by the United States Government.

DISPOSITION INSTRUCTIONS

Destroy this report when it is no longer needed.
Do not return it to the originator.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER MTL TR 90-51	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE *(and Subtitle)* HOT ISOSTATIC PRESSING OF SUPERCONDUCTING CERAMICS		5. TYPE OF REPORT & PERIOD COVERED Final Report 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR*(s)* Kerry T. Richards and CPT Richard H. Benfer		8. CONTRACT OR GRANT NUMBER*(s)*
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001 SLCMT-EMC		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Laboratory Command 2800 Powder Mill Road Adelphi, Maryland 20783-1145		12. REPORT DATE October 1990 13. NUMBER OF PAGES 19
14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)*		15. SECURITY CLASS. *(of this report)* Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

Ceramic materials
Hot isostatic pressing (HIP)
Superconducting materials
Oxygen stoichiometry
Physical properties

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

(SEE REVERSE SIDE)

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

Block No. 20

ABSTRACT

Hot isostatic pressing (HIP) was studied as a method for processing bulk superconductors. Superconducting powder was derived from the calcination of nitrated Y_2O_3, CuO and $BaCO_3$ powder. The powder was HIPed using pressures of 70, 140, and 210 MPa with temperatures of 820°C and 950°C for a hold time of 1 hour. The density, hardness and Young's modulus of HIPed samples were higher than those of sintered control samples. Superconducting transition temperatures >92°K were achieved without post-HIP annealing of the samples. Keywords: Superconductors/ ceramic materials; Sintering; Transition temperature; Modulus of elasticity; Encapsulation; Hot pressing; Isostatic pressing.
(MM)

CONTENTS

BACKGROUND

Much work has been devoted to high temperature ceramic superconductors since their recent discovery.[1,2] Most advances have come in the area of thin films where prototype applications have been developed and tested. The processing of bulk shapes (e.g., bars, rods, rings) for use as components in larger systems (e.g., motors, rail guns) has progressed more slowly.

Hot isostatic pressing (HIP) is a method of fabricating bulk ceramic shapes through the application of high pressures and temperatures. The HIP process can readily consolidate complex ceramic shapes at lower temperatures than conventional sintering. Currently, HIP units can operate at pressures over 200 MPa and temperatures in excess of 2000°C. The pressure is applied by a high pressure gas which is commonly nitrogen or argon. More recently, oxygen/inert gas mixtures have been used.

A material must have closed porosity in order to be successfully HIPed without encapsulation. In other cases, the material must be sealed in a capsule which acts as a pressure transfer membrane. The capsule material must be able to withstand the high temperatures and pressures found in the HIP chamber. In addition, it must have the flexibility to conform to the sample material. Capsules are generally made of metals and glasses.

HIPing has been used to fabricate new high temperature superconductors.[3-6] Tien et al.[3] HIPed $YBa_2Cu_3O_{7-x}$ using copper cans at 100 MPa and temperatures of 750°C and 900°C. The resulting material was 99.3% dense and after heat treatment in air the material exhibited Meissner exclusion. Meissner exclusion indicated the presence of a superconducting phase. They note, however, that it is preferable to produce the final oxygen stoichiometry of the ceramic in the as-HIPed state.

Other researchers have also noted the requirement of an oxygen anneal after HIPing in order to regain superconducting properties. Snow et al.[4] HIPed the $YBa_2Cu_3O_{7-x}$ material in stainless steel cans at 103 MPa and 850°C to over 95% of theoretical density. An oxygen anneal at 850°C for 2 hours was required for superconducting properties to appear. Meissner exclusion at low temperatures approached 100%.

Sadananda et al.[5] studied HIPing superconductors in both glass (Pyrex) and metal (stainless steel) tubes. HIPing at 850°C and 207 MPa for 1 hour in the Pyrex tube resulted in a material that became superconducting at 65°K without anneal. This group also noted a grain size refinement where the average grain size in the compact decreased after HIPing. They propose that the PdV work done during HIPing caused the fracture of brittle ceramic oxide particles. The low transition temperature (T_c) of 65°K is attributed to oxygen loss occurring during vacuum encapsulation or during pressing.

1. BEDNORZ, J. G. and MULLER, K. A. *Possible High-T_c Superconductivity in the Ba-La-Cu-O System.* Z. Phys. B., v. 64, 1986, p. 189-193.
2. WU, M. K. et al. *Superconductivity at 93°K in a New Mixed Phase Y-Ba-Cu-O Compound System at Ambient Pressure.* Phys. Rev. Lett., v. 58, 1987, p. 908.
3. TIEN, J. K., HENDRIX, B. C., BOROFKA, J. C., and ABE, T. *Hot Isostatic Pressing (HIP) for the Densification of Oxide Superconductors.* Mat. Res. Soc. Spring Meeting, 1988.
4. SNOW, D. B., WEINBERGER, B. R., PETERSON, G. G., LYNDS, L., EASTON, H., BURILA, C. T., POTREPKA, D. M., and KUWABARA, M. *Processing, Microstructure and Properties of $YBa_2Cu_3O_{7-\delta}$/Ag Superconductors.* Proceedings of TMS, March 1989.
5. SADANANDA, K., SINGH, J. K., IMAN, M. A., OSOFSKY, M., LETOURNEAU, V., and RICHARDS, L. E. *Effect of Hot Isostatic Pressing on $RBa_2Cu_3O_7$ Superconductors.* Advanced Ceramic Materials, v. 3, no. 5, 1988, p. 524-526.

Niska et al.[6] have noted that HIPing of the $YBa_2Cu_3O_{7-x}$ can lead to oxygen loss and a resulting orthorhombic-to-tetragonal phase transformation. This reversible structural transformation occurs with changing oxygen stoichiometry, going from orthorhombic at x = 0 to tetragonal at x = 1.0.[7] They also noted that glass encapsulated material retained the superconducting orthorhombic phase while the stainless steel encapsulated material became tetragonal. They explain that oxygen is evolved by the superconducting material while it decomposes during heating. This oxygen may more readily diffuse out through the metal, and more importantly, the oxygen can react with the metal canister to form an oxide. A glass encapsulated system, therefore, results in a higher equilibrium partial pressure of oxygen which favors the retention of the orthorhombic phase. However, this higher oxygen pressure can also oppose the densification process. Therefore, processing conditions must be carefully chosen in order to maximize both T_c and density.

The goal of this research was to develop a HIP process for densifying $YBa_2Cu_3O_{7-x}$ while simultaneously retaining high temperature T_c's. This would eliminate the need for a post-HIP oxygen anneal step. The encapsulation method was designed to adjust the oxygen equilibrium in the samples through the addition of an oxygen donor material. This material would decompose and evolve oxygen in order to increase the oxygen partial pressure. BaO_2 was chosen as the oxygen donor for this work because of its compatibility with the $YBa_2Cu_3O_{7-x}$ system. With the appropriate choice of process conditions, it was expected that fully dense $YBa_2Cu_3O_{7-x}$ could be HIPed while still retaining its superconducting properties.

An alternative method for controlling oxygen stoichiometry is accomplished by the recently developed oxygen HIP. Kobe Steel of Japan, for instance, has developed a HIP unit that uses an oxygen/inert gas mixture and plans to use it for their superconductor processing research.[8] However, due to the problems associated with high pressure oxygen at high temperatures, the oxygen HIP unit is more complex and expensive than a standard HIP unit. The method outlined in this report is inexpensive and readily adopted.

EXPERIMENTAL

Powder Preparation

The starting superconducting powder was prepared by the solid-state reaction of Y_2O_3, $BaCO_3$ and CuO powders. Figure 1 illustrates a flowchart of the powder processing sequence. The powders were mixed in stoichiometric ratio, reacted with nitric acid and dried at 150°C. The nitrated powder was reacted at 600°C in air for 20 hours and then ground using a mortar and pestle. The powder was then calcined in air twice, with an intermediate grinding, at 940°C for 6 hours. The resulting $YBa_2Cu_3O_{7-x}$ powder was then heated to 500°C and cooled to 275°C over 12 hours in order to maximize the oxygen content by the compound. The powder was examined by an X-ray diffractometer (Diano Model 8535) using Cu-Kα radiation.

6. NISKA, J., LOBERG, B., and EASTERLING, K. *Effect of Oxygen Loss on Densification When Hot Isostatic Pressing $YBa_2Cu_3O_{7-x}$.* J. Am. Ceram. Soc., v. 72, no. 8, 1989, p. 1508-1510.
7. GALLAGHER, P. K., O'BRYAN, H. M., SUNSHINE, S. A., and MURPHY, D. W. *Oxygen Stoichiometry in $Ba_2YCu_3O_x$.* Mat. Res. Bull., v. 22, 1987, p. 995-1006.
8. *Oxygen HIP for Superconducting Ceramics Research Fabrication.* Nebea Keizai Shimbunsha, January 24, 1988, p. 4.

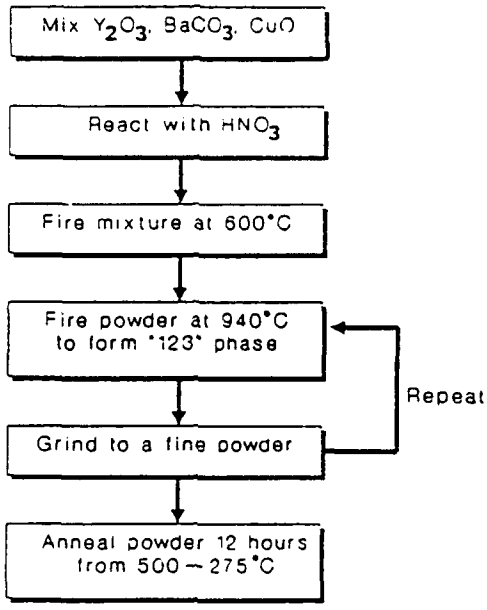

Figure 1. Flowchart of the powder processing steps used for producing single phase $YBa_2Cu_3O_{7-x}$ material.

Sample Preparation

Powder samples were cold isostatically pressed (CIPed) in flexible molds at 160 MPa. The resulting sample size was approximately 13 mm long x 9 mm diameter and weighed 4 g. Both sintered and HIPed samples were produced from the same batch of $YBa_2Cu_3O_{7-x}$ powder. The sintered samples were used for comparison purposes. Table 1 outlines the processing conditions used in both sintering and HIPing.

Table 1. PROCESSING CONDITIONS

	Temperature (°C)	Pressure (MPa)	Time (Hrs.)
Sinter	960	Atmospheric	6
HIP	820	69	1
		138	1
		207	1
	950	69	1
		138	1
		207	1

Sintered samples were fired in air at 960°C for 6 hours. A slow anneal in air from 500°C to 200°C over 6 hours was used to maximize the oxygen content of the samples.

Two types of HIP sample configurations were used. Both types were vacuum-sealed in Pyrex capsules. The type A configuration consisted of a pressed superconducting powder sample surrounded by silica cloth. Type B consisted of a pressed powder sample and 1 g of BaO_2 surrounded by silica cloth.

HIPing was performed in an Autoclave Engineers 30M hot isostatic press using N_2 gas and a graphite furnace. Six temperature/pressure conditions for HIPing were chosen (T = 820°C and 950°C; P = 69, 138 and 207 MPa). Four encapsulated samples were HIPed during each run; two of type A and two of type B. Samples were held at temperature and pressure for 1 hour, after which the furnace was shut off and the samples allowed to cool. Figure 2 illustrates typical pressure and temperature schedule for a 138 MPa and 820°C HIP run.

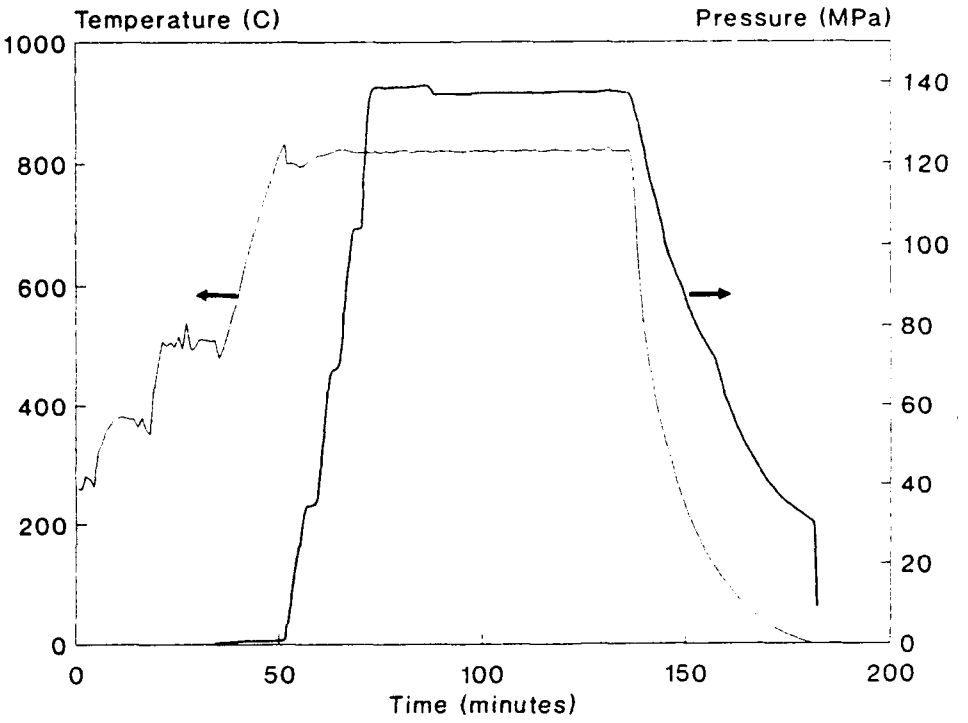

Figure 2. Pressure and temperature measurements for a 138 MPa and 820°C HIP run. Note that heat is applied first in order to soften the encapsulating glass. Hold time at temperature and pressure is 60 minutes.

Samples were removed from the glass capsule and then sliced into sections with a diamond saw. Measurements were performed on these sliced sections. Sample sections used for porosity measurements and hardness testing were mounted and polished in kerosene. All materials were stored in a vacuum desiccator.

Characterization

Samples were characterized for (1) density, (2) porosity, (3) Young's Modulus, (4) Knoop hardness, (5) crystalline phases and (6) T_c.

Bulk density was measured by the Archimedes method using distilled water. Porosity measurements were made in accordance with ASTM E 562-83 (Standard Practice for Determining Volume Fraction by Systematic Manual Point Count). The point count was made using a scanning electron microscope at 500X magnification and an 81 point grid. One hundred fields were counted for each sample.

Young's modulus was determined by ultrasound longitudinal and shear wave measurements using 6.4 mm diameter, 5 MHz longitudinal and shear wave transducers. Hardness testing was performed with the Knoop hardness tester using a 100 g load.

Sliced sections were ground for powder X-ray diffraction. X-ray patterns were generated with an X-ray diffractometer (Diano Model 8535) using Cu-Kα radiation. Phase identification was determined by comparison with JCPDS cards and published patterns.*†

Transition temperatures were determined through the alternate current (a.c.) magnetization tests. Samples cut to approximately 2 mm x 2 mm x 5 mm were used. Measurements were made by placing the samples within concentric coils driven by a 20 kHz, 1 volt rms source (HP651B Test Oscillator). A lock-in amplifier (EG&G Model 128A) was used to filter and amplify the pick-up signal. Temperature was measured through a Type K thermocouple referenced to 0°C. Critical temperatures were determined by noting the abrupt change in the output voltage which corresponds to the onset of diamagnetism in the sample. Measurements were made as the sample was warmed through the T_c. Figure 3 shows a schematic diagram of the measuring system.

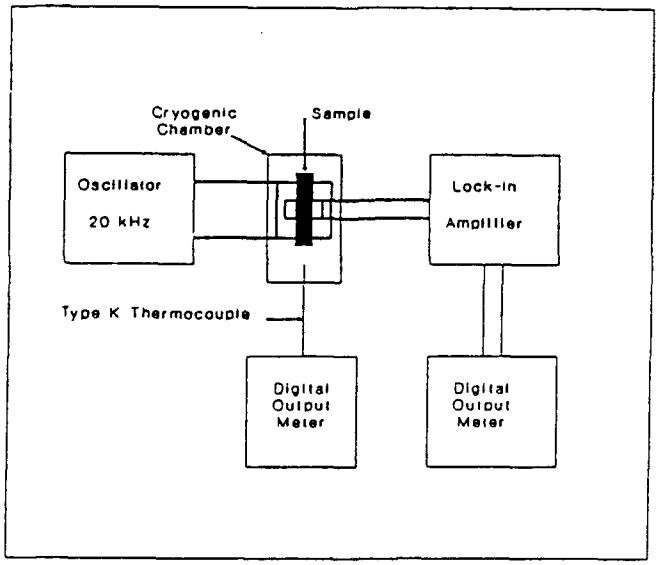

Figure 3. Diagram of the transition temperature measurement system. Cryogenic chamber uses liquid nitrogen as a coolant.

*Barium Copper Yttrium Oxide, $Ba_2Cu_3YO_7$. Powder Diffraction, v. 2, 1987, p. 192.
†Barium Copper Yttrium Oxide, $BaCuT_2O_5$. Powder Diffraction, v. 2, 1987, p. 192.

RESULTS AND DISCUSSION

Encapsulation

The encapsulation method proved to be relatively simple and reliable. The silica glass cloth separated the sample from the BaO_2 and from the capsule. It also helped prevent damage to the green body while sealing the capsule. Cooling the samples quickly from the HIPing temperature normally caused the encapsulating glass to crack, making removal of the sample from the capsule a simple matter of peeling away the cracked glass. Samples HIPed at 950°C were normally more difficult to remove from the capsule than samples HIPed at 850°C.

Phase Identification

The X-ray diffraction pattern of starting superconducting powders was compared to published patterns*† and identified as a single phase composition of orthorhombic $YBa_2Cu_3O_{7-x}$. The theoretical density of this phase is 6.383 g/cm^3.

The decomposition of barium carbonate presents the greatest problems during the calcination process.[9] Significant amounts of $BaCO_3$ can remain up to 1200°C.[10] Therefore, nitrated precursors were used in this work. No residual $BaCO_3$ was detected in X-ray diffraction patterns of the starting powder. These results may be explained by the difference in decomposition temperatures for barium carbonate and barium nitrate (1450°C versus 600°C).[11] The nitrate yields a more complete reaction at lower temperatures.

X-ray diffraction patterns of the samples HIPed at 820°C were identified as an orthorhombic $YBa_2Cu_3O_{7-x}$ phase. Residual amounts of orthorhombic Y_2BaCuO_5 phase ("211"), CuO, and $Ba(OH)_2$, were also detected and are highlighted on the X-ray pattern in Figure 4. These phases have been shown to be a product of the following decomposition reaction[12] with water:

$$2YBa_2Cu_3O_7 + 3H_2O \rightarrow Y_2BaCuO_5 + 5CuO + 3Ba(OH)_2 + 1/2O_2 \quad (1)$$

This reaction takes place slowly with humid air at room temperature and rapidly in air at 85°C and 85% relative humidity.[13] Therefore, the "211" phase may have developed during cutting, storage, or during preparation of the X-ray samples.

*Barium Copper Yttrium Oxide, $Ba_2Cu_3YO_7$. Powder Diffraction, v. 2, 1987, p. 192.

†Barium Copper Yttrium Oxide, $BaCuT_2O_5$. Powder Diffraction, v. 2, 1987, p. 192.

9. YAN, M. F., LING, H. C., O'BRYAN, H. M., GALLAGHER, P. K., and RHODES, W. W. *Process-related Problems of $YBa_2Cu_3O_x$ Superconductors.* Mat. Sci. and Eng., v. B1, 1988, p. 119-129.
10. NAVEH, J. and PELLY, I. *On the Preparation of $YBa_2Cu_3O_{7-x}$ Ceramic High-Temperature Superconductor.* Mat. Res. Bull., v. 24, 1989, p. 282-287.
11. TARASCON, J. M., BARBOUX, P. B., BAGLEY, B. G., GREENE, L. H., and HULL, G. W. *On Synthesis of High T_c Superconducting Perovskites.* Mat. Sci. and Eng., v. B1, 1988, p. 29-36.
12. YAN, M. F., BARNS, R. L., O'BRYAN, Jr., H. M., GALLAGHER, P. K., SHERWOOD, R. C., and JIN, S. Water Interaction with the Superconducting $YBa_2Cu_3O_7$ Phase. Appl. Phys. Lett., v. 51, 1987, p. 532.
13. BARNS, R. L. and LAUDISE, R. A. *Stability of Superconducting $YBa_2Cu_3O_7$ in the Presence of Water.* Appl. Phys. Lett., v. 51, no. 17, 1987, p. 1373-1375.

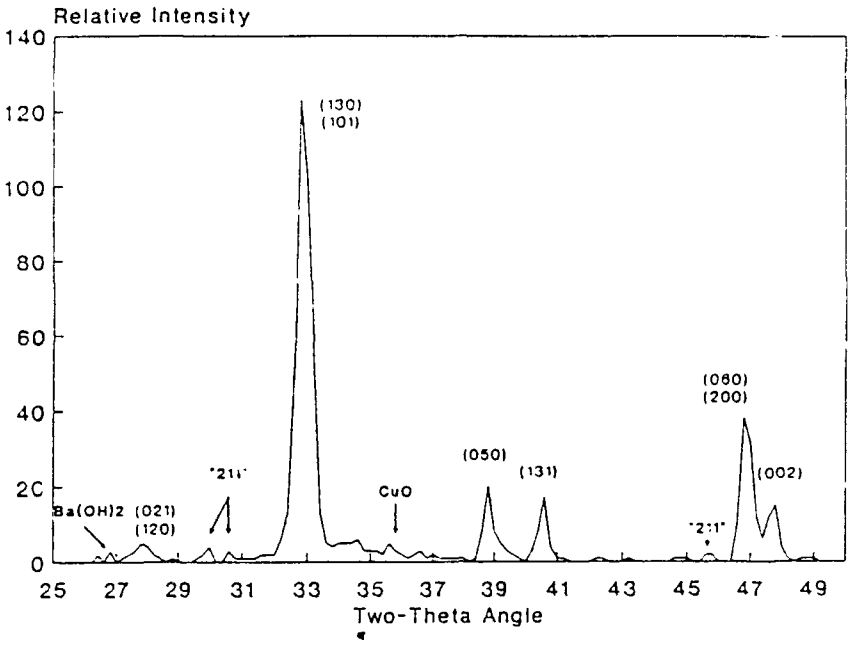

Figure 4. X-ray diffraction pattern for a sample HIPed at 69 MPa and 820°C with BaO_2. The (hkl) planes corresponding to the major peaks for $YBa_2Cu_3O_7$ are identified. Note also the peaks attributed to the "211" phase, CuO and $Ba(OH)_2$.

Physical Properties

The density data is plotted in Figures 5a and 5b. Samples show an increase in the density with an increase in HIP pressure. Samples HIPed at 820°C have higher densities than those HIPed at 950°C. For each temperature, type B samples had lower densities than type A samples.

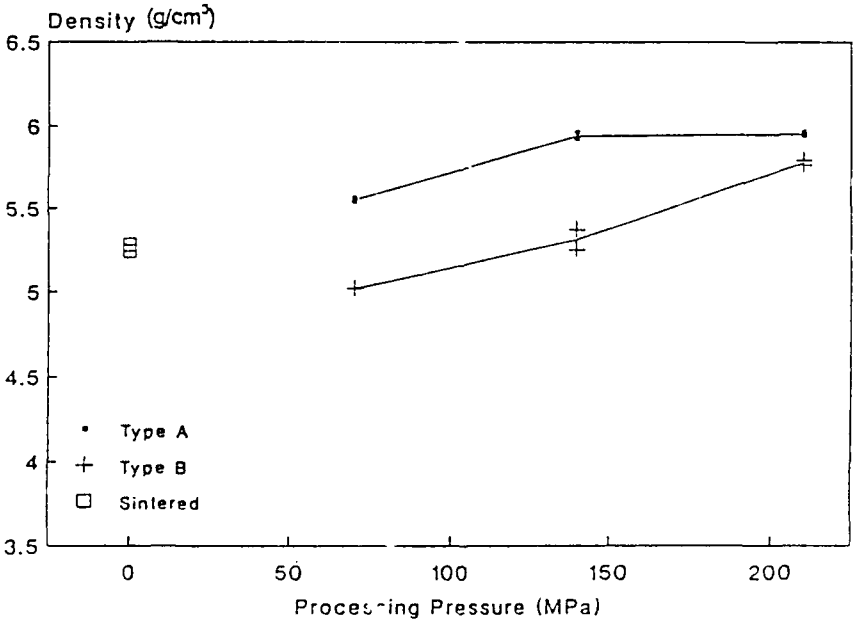

Figure 5a. Density measurement results for samples HIPed at 820°C.

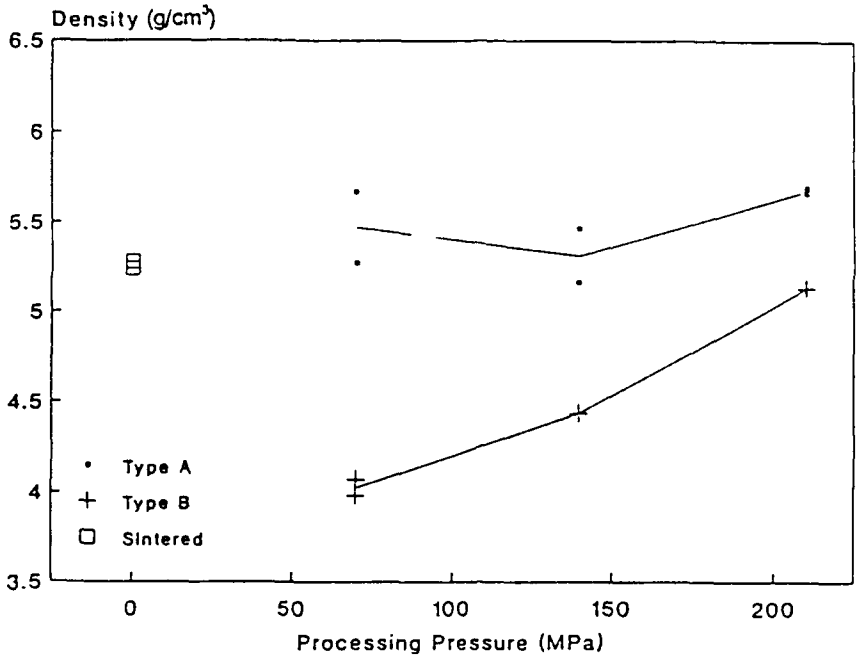

Figure 5b. Density measurement results for samples HIPed at 950°C.

Results of porosity measurements are shown in Figures 6a and 6b. Porosity decreased with increasing HIP pressure. Samples HIPed at 820°C have less porosity than those HIPed at 950°C. For a given HIP temperature, type B samples had greater porosity than type A samples. As expected, decreasing porosity was found to correlate well with increasing density (see Figure 6c).

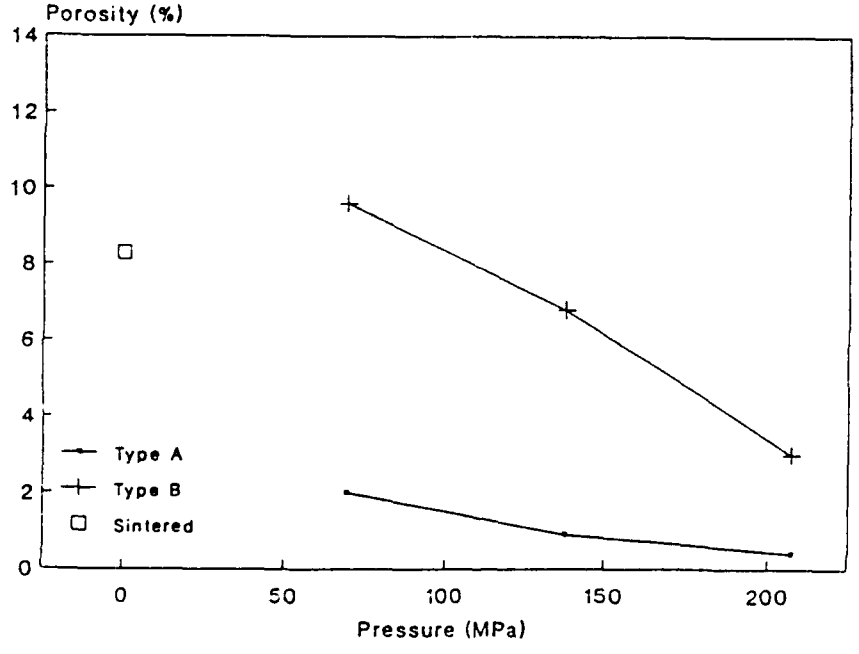

Figure 6a. Porosity measurements for samples HIPed at 820°C.

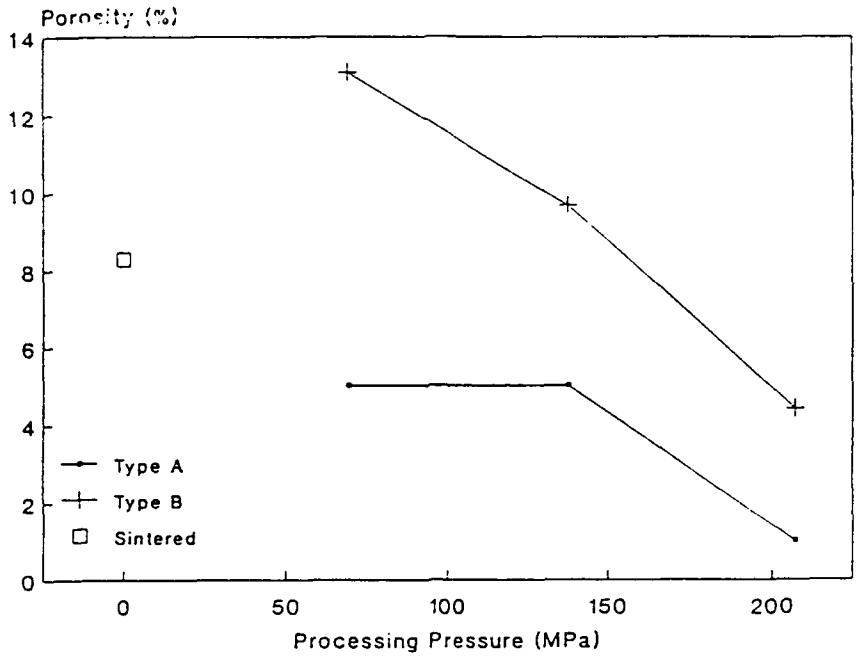

Figure 6b. Porosity measurements for samples HIPed at 950°C.

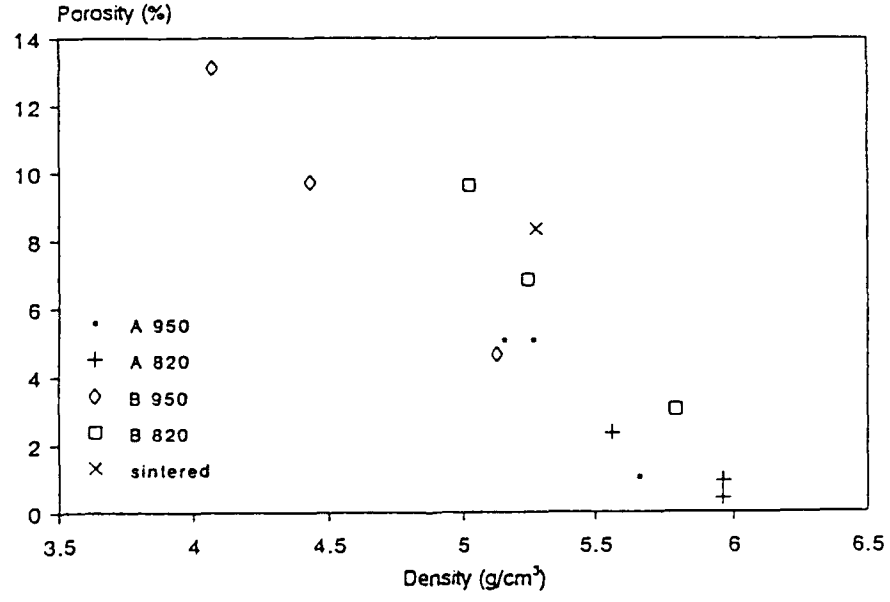

Figure 6c. Porosity versus density correlation for all samples.

Young's modulus results are presented in Figures 7a and 7b. The modulus increased with increasing pressure. Samples HIPed at 820°C had higher modulus values than those HIPed at 950°C. For a given HIP temperature, type B samples had lower modulus values than type A samples. Figure 7c shows the trend of increasing Young's modulus with higher density.

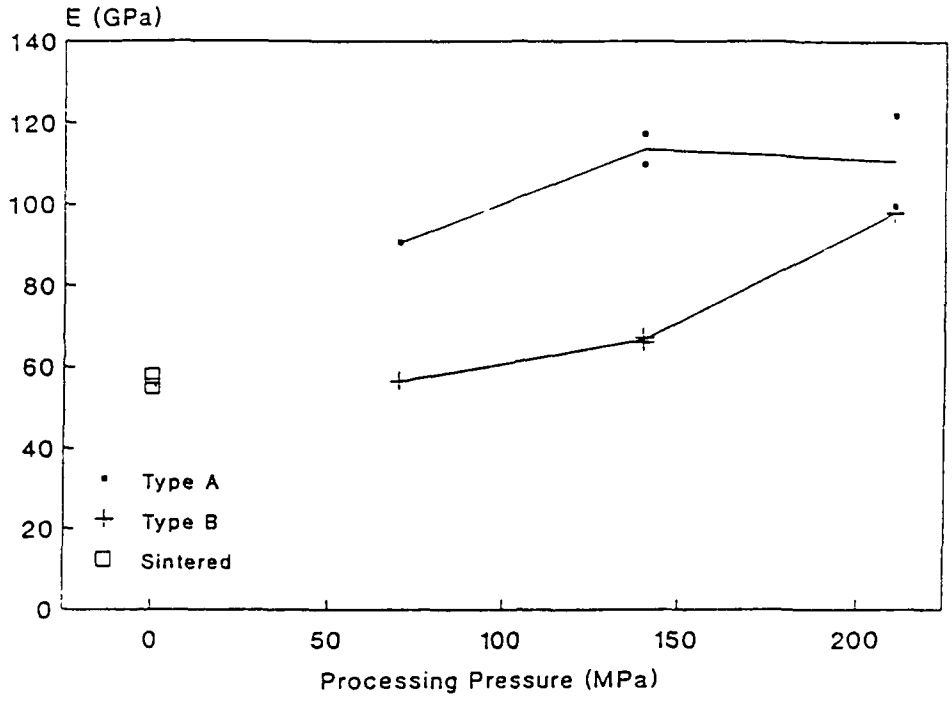

Figure 7a. Young's modulus results for samples HIPed at 820°C.

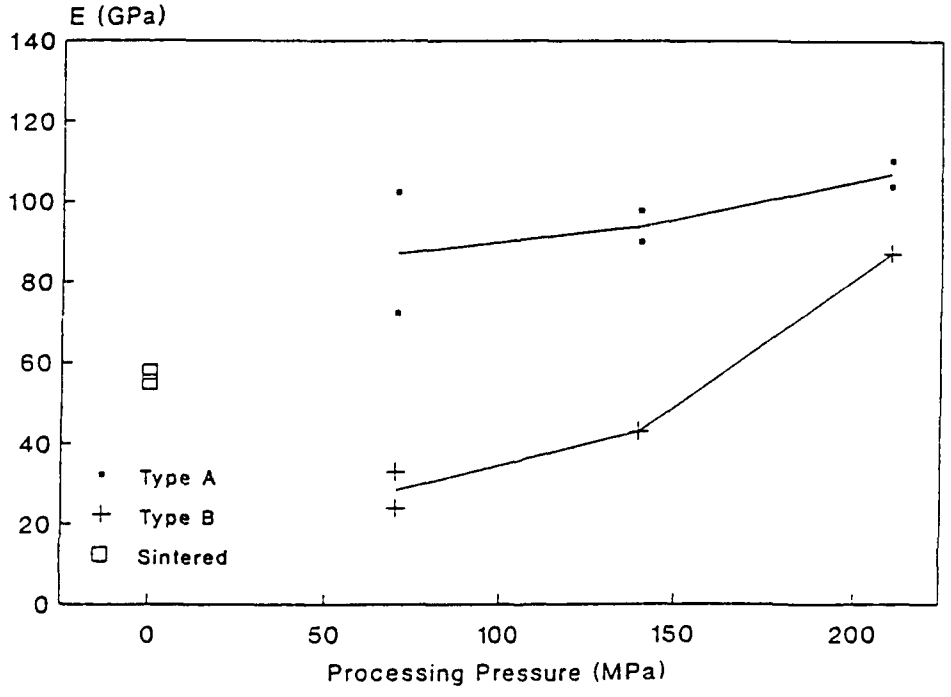

Figure 7b. Young's modulus results for samples HIPed at 950°C.

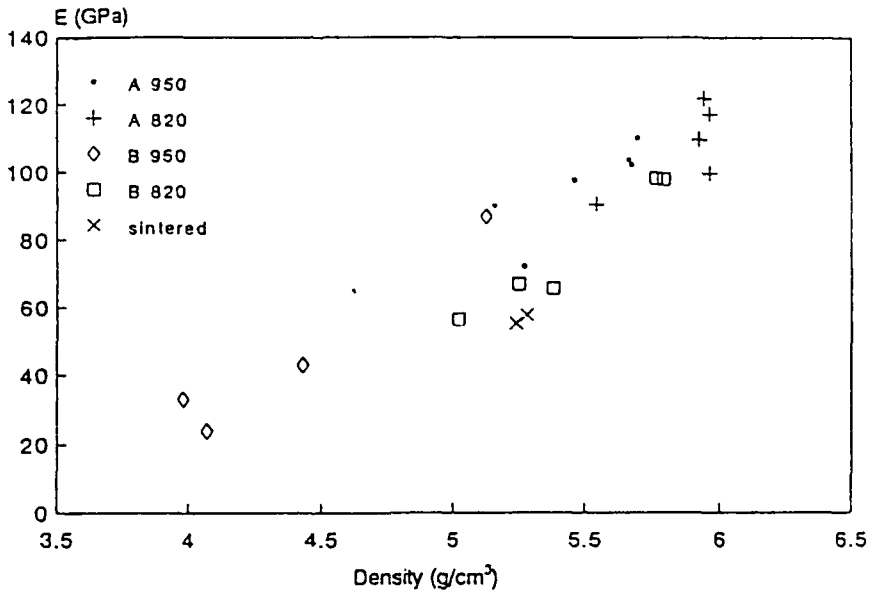

Figure 7c. Young's modulus versus density correlation for all samples.

Figures 8a and 8b show the Knoop hardness data. The hardness increased with increasing HIP pressure. Samples HIPed at 820°C had a higher hardness than those HIPed at 950°C. For each temperature, type B samples had lower hardness values than type A samples. Figure 8c indicates that hardness rises with increasing density of the sample.

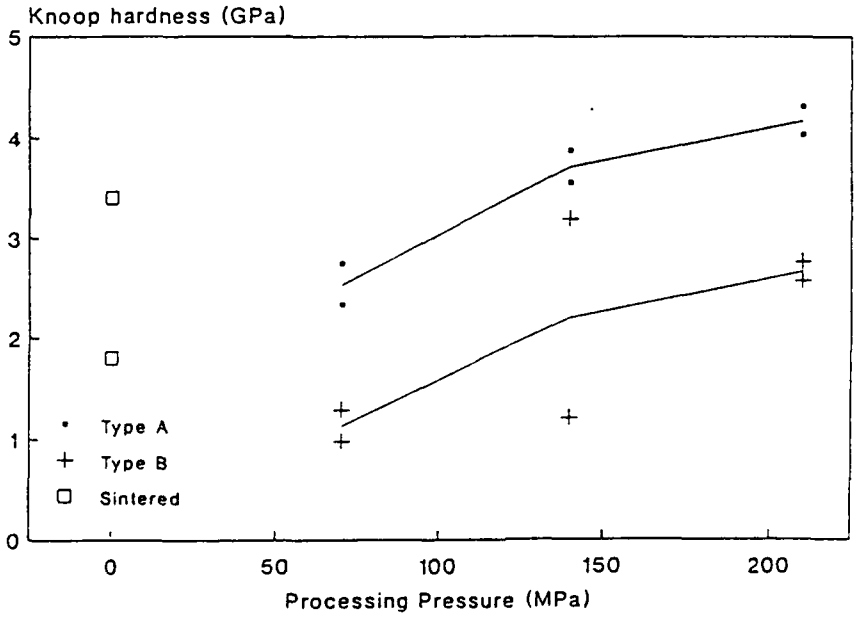

Figure 8a. Knoop hardness results for samples HIPed at 820°C.

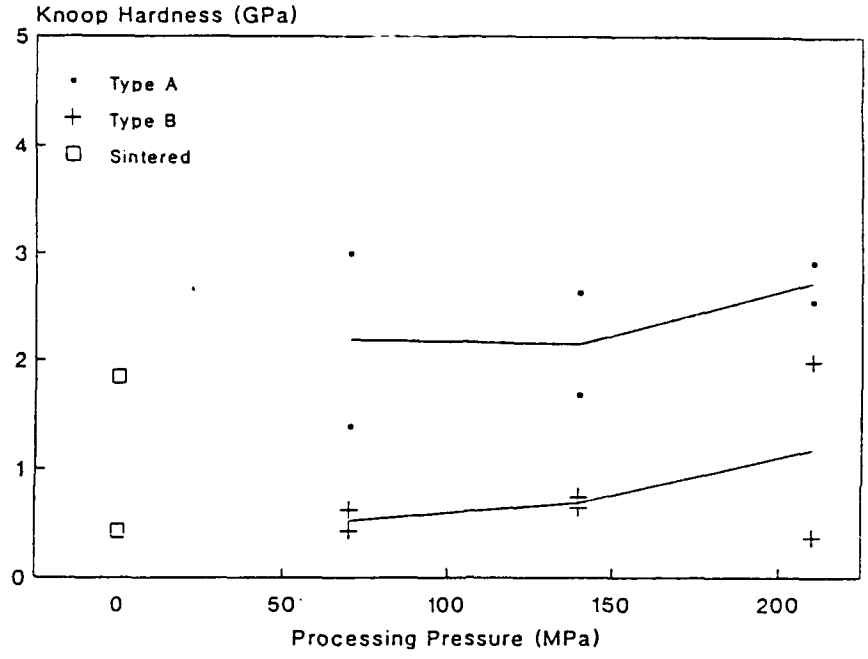

Figure 8b. Knoop hardness results for samples HIPed at 950°C.

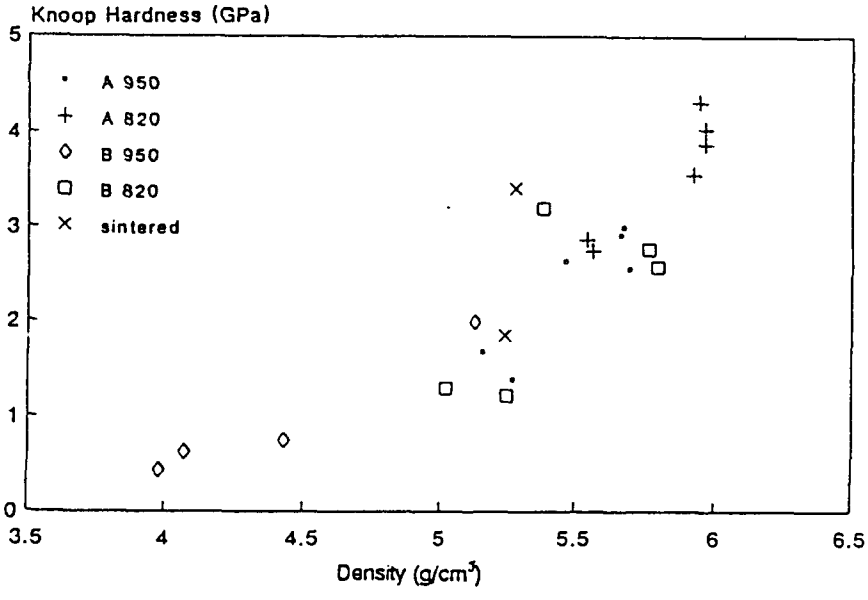

Figure 8c. Knoop hardness versus density correlation for all samples.

The following explanation of the above trends is proposed. All samples were sealed under vacuum in glass tubes. During the initial sequence of the HIP cycle, the samples were heated to the soak temperature (820°C and 950°C) with the HIP chamber under vacuum or very low pressure. Figure 2 illustrates these conditions, which allowed oxygen from the

samples in the tube to dissociate. The evolved gas became entrained in the sample, inhibiting pore closure during HIPing. Samples heated to 950°C generated more oxygen gas, contributing to their higher porosity and lower density. The lower densities of these samples then resulted in poorer physical properties (hardness, modulus).

Comparison of the HIPed samples with the sintered control samples shows that an improvement in the physical properties was attained using HIPing. Properties are most enhanced for samples HIPed at 820°C and 207 MPa. Under those conditions, HIPed samples had significantly better properties than the sintered samples. An overall evaluation shows that HIPing samples at 820°C and 207 MPa with BaO_2 produced samples with good physical and superconducting properties.

Transition Temperatures

A typical example of the trace derived from a.c. magnetization measurements is shown in Figure 9. The ordinate is shown in arbitrary units while the abscissa is in degrees Kelvin. The sharp bend in the curve is indicative of diamagnetic shielding and defines the T_c of the material. Use of this magnetic transition, instead of the resistive transition, offers two distinct advantages. First, no leads or connections must be made, making the measurement non-intrusive. Second, this method avoids the spurious results of resistive measurements caused by lead connections and grain boundary effects.

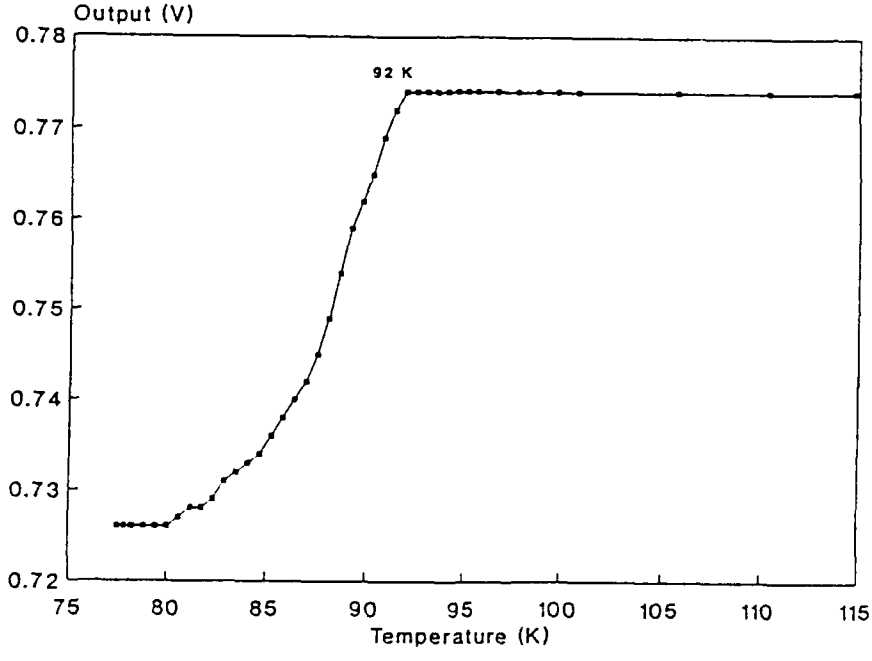

Figure 9. A.C. magnetization trace for a sintered sample. The sharp bend in the curve indicates the T_c.

Figure 10a shows the T_c of samples HIPed at 820°C. Type B samples recorded higher T_c's than type A samples. The difference in T_c varied between 6 and 12 degrees Kelvin. The T_c's of the type B samples compared favorably with those of the sintered control specimens.

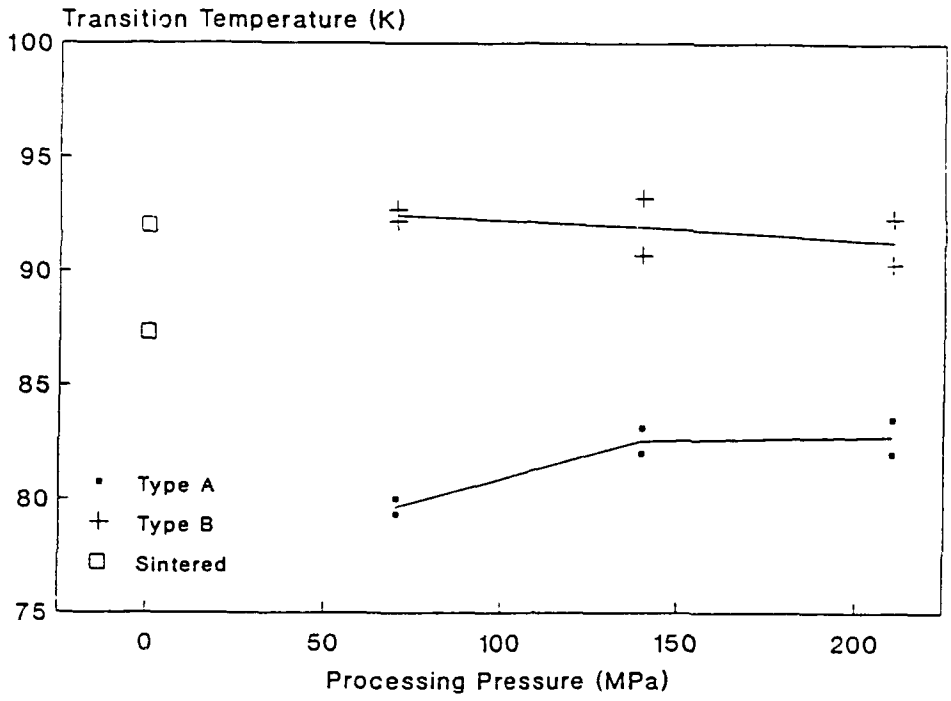

Figure 10a. T_C for samples HIPed at 820°C.

The release of oxygen from the BaO_2 at elevated temperatures establishes a higher oxygen partial pressure within the capsule. The higher T_c's for the type B samples suggests that this environment promotes a higher final oxygen content in the HIPed sample. This hypothesis agrees with reported literature which finds that the T_c increases with increasing oxygen content of the ceramic.[14]

An overall evaluation shows that samples HIPed with BaO_2 at 820°C and 207 MPa possessed good physical properties and high superconducting T_c's. These characteristics are equal to or surpassing those of sintered samples. In addition, these properties are obtained at a processing temperature 140°C lower than that used for sintering.

Figure 10b shows the T_c's of samples HIPed at 950°C. These results are even lower than those for samples HIPed at 820°C. Some T_c's were below 77°K and could not be measured by the test system used in this work.

In $YBa_2Cu_3O_{7-x}$, T_c is maximized at $x = 0$ and it decreases with the decreasing oxygen content of the material.[14] Therefore, any processing step which leads to decreased oxygen content in the material will lower that material's T_c. At 950°C, both a greater dissociation of $YBa_2Cu_3O_{7-x}$ and an increased loss of oxygen through the encapsulant material occur. It is proposed that these processes lead to a decreased oxygen content in the HIPed samples and results in lower T_c's for these samples.

14. MURPHY, D. W., JOHNSON, Jr., D. W., JIN, S., and HOWARD, R. E. *Processing Technologies for the 93°K Superconductor $Ba_2YCu_3O_7$*. Science, v. 241, August 19, 1988, p. 923.

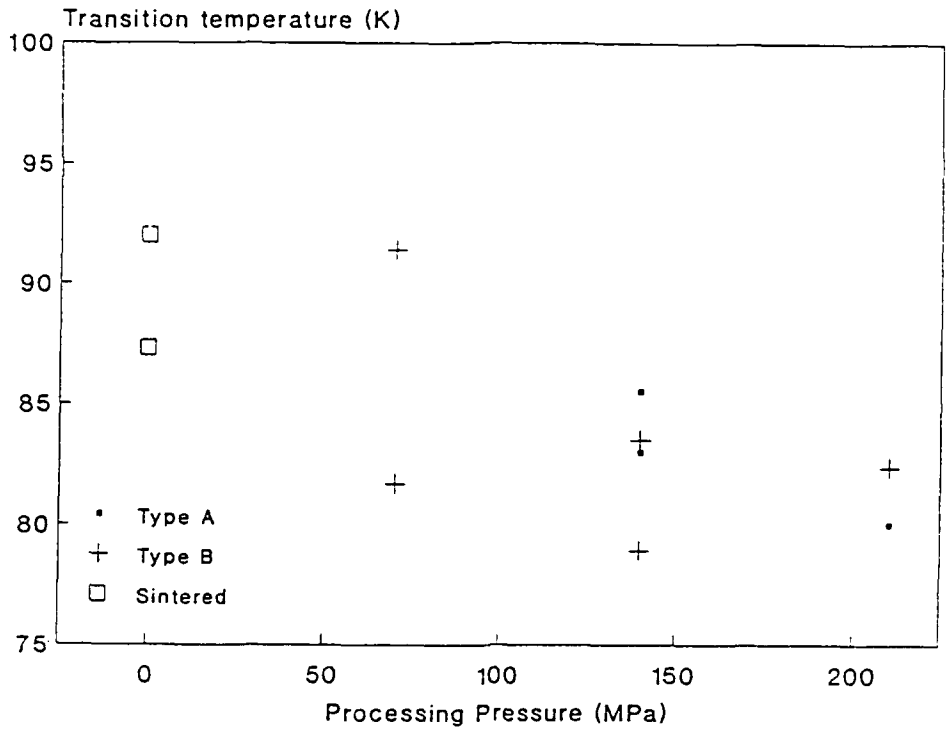

Figure 10b. T_C for samples HIPed at 950°C.

SUMMARY

HIPing of the ceramic superconductor $YBa_2Cu_3O_{7-x}$ was found to be a simple and reliable method for the consolidation of bulk shapes. The following observations are made:

- HIPing can produce samples with better physical properties at lower processing temperatures than conventional sintering. These improved physical properties are shown to correlate well with higher densities.
- The addition of the BaO_2 oxidant allows for HIPed samples to retain high T_c's. Samples HIPed at 820°C with BaO_2 had T_c's greater than 92°K.
- Unlike results reported previously in the literature, these T_c's were achieved in the final shape without a post-HIP anneal.
- The careful tailoring of HIP processing conditions (temperature, pressure, encapsulant and oxidant) can result in samples produced with both enhanced physical properties and retained superconducting properties.

ACKNOWLEDGMENTS

The authors thank L. Tardiff for ultrasonic measurements and Young's Modulus determination, R. Lancto for Knoop hardness measurements and R. Hinxman for X-ray diffraction work.

REFERENCES

1. BEDNORZ, J. G. and MULLER, K. A. *Possible High-T_c Superconductivity in the Ba-La-Cu-O System.* Z. Phys. B., v. 64, 1986, p. 189-193.
2. WU, M. K. et al. *Superconductivity at 93 K in a New Mixed Phase Y-Ba-Cu-O Compound system at Ambient Pressure.* Phys. Rev. Lett., v. 58, 1987, p. 908.
3. TIEN, J. K., HENDRIX, B. C., BOROFKA, J. C., and ABE, T. *Hot Isostatic Pressing (HIP) for the Densification of Oxide Superconductors.* Mat. Res. Soc. Spring Meeting, 1988.
4. SNOW, D. B., WEINBERGER, B. R., PETERSON, G. G., LYNDS, L., EASTON, H., BURILA, C. T., POTREPKA, D. M., and KUWABARA, M. *Processing, Microstructure and Properties of $YBa_2Cu_3O_{7-\delta}/Ag$ Superconductors.* Proceedings of TMS, March 1989.
5. SADANANDA, K., SINGH, A. K., IMAN, M. A., OSOFSKY, M., LETOURNEAU, V., and RICHARDS, L. E. *Effect of Hot Isostatic Pressing on $RBa_2Cu_3O_7$ Superconductors.* Advanced Ceramic Materials, v. 3, no. 5, 1988, p. 524-526.
6. NISKA, J., LOBERG, B., and EASTERLING, K. *Effect of Oxygen Loss on Densification When Hot Isostatic Pressing $YBa_2Cu_3O_{7-\delta}$.* J. Am. Ceram. Soc., v. 72, no. 8, 1989, p. 1508-1510.
7. GALLAGHER, P. K., O'BRYAN, H. M., SUNSHINE, S. A., and MURPHY, D. W. *Oxygen Stoichiometry in $Ba_2YCu_3O_x$.* Mat. Res. Bull., v. 22, 1987, p. 995-1006.
8. *Oxygen HIP for Superconducting Ceramics Research Fabrication.* Nebea Keizai Shimbunsha, January 24, 1988, p. 4.
9. YAN, M. F., LING, H. C., O'BRYAN, H. M., GALLAGHER, P. K., and RHODES, W. W. *Process-related Problems of $YBa_2Cu_3O_x$ Superconductors.* Mat. Sci. and Eng., v. B1, 1988, p. 119-129.
10. NAVEH, J. and PELLY, I. *On the Preparation of $YBa_2Cu_3O_{7-x}$ Ceramic High-Temperature Superconductor.* Mat. Res. Bull., v. 24, 1989, p. 282-287.
11. TARASCON, J. M., BARBOUX, P. B., BAGLEY, B. G., GREENE, L. H., and HULL, G. W. *On Synthesis of High T_c Superconducting Perovskites.* Mat. Sci. and Eng., v. B1, 1988, p. 29-36.
12. YAN, M. F., BARNS, R. L., O'BRYAN, Jr., H. M., GALLAGHER, P. K., SHERWOOD, R. C., and JIN, S. Water Interaction with the Superconducting $YBa_2Cu_3O_7$ Phase. Appl. Phys. Lett., v. 51, 1987, p. 532.
13. BARNS, R. L. and LAUDISE, R. A. *Stability of Superconducting $YBa_2Cu_3O_7$ in the Presence of Water.* Appl. Phys. Lett., v. 51, no. 17, 1987, p. 1373-1375.
14. MURPHY, D. W., JOHNSON, Jr., D. W., JIN, S., and HOWARD, R. E. *Processing Technologies for the 93K Superconductor $Ba_2YCu_3O_7$.* Science, v. 241, August 19, 1988, p. 923.

DISTRIBUTION LIST

No. of Copies	To
1	Office of the Under Secretary of Defense for Research and Engineering, The Pentagon, Washington, DC 20301
	Commander, U.S. Army Laboratory Command, 2800 Powder Mill Road, Adelphi, MD 20783-1145
1	ATTN: AMSLC-IM-TL
1	AMSLC-CT
	Commander, Defense Technical Information Center, Cameron Station, Building 5, 5010 Duke Street, Alexandria, VA 22304-6145
2	ATTN: DTIC-FDAC
1	Metals and Ceramics Information Center, Battelle Columbus Laboratories, 505 King Avenue, Columbus, OH 43201
	Commander, Army Research Office, P.O. Box 12211, Research Triangle Park, NC 27709-2211
1	ATTN: Information Processing Office
	Commander, U.S. Army Materiel Command, 5001 Eisenhower Avenue, Alexandria, VA 22333
1	ATTN: AMCLD
	Commander, U.S. Army Materiel Systems Analysis Activity, Aberdeen Proving Ground, MD 21005
1	ATTN: AMXSY-MP, H. Cohen
	Commander, U.S. Army Missile Command, Redstone Scientific Information Center, Redstone Arsenal, AL 35898-5241
1	ATTN: AMSMI-RD-CS-R/Doc
1	AMSMI-RLM
	Commander, U.S. Army Armament, Munitions and Chemical Command, Dover, NJ 07801
2	ATTN: Technical Library
1	AMDAR-LCA, Mr. Harry E. Pebly, Jr., PLASTEC, Director
	Commander, U.S. Army Natick Research, Development and Engineering Center, Natick, MA 01760-5010
1	ATTN: Technical Library
	Commander, U.S. Army Satellite Communications Agency, Fort Monmouth, NJ 07703
1	ATTN: Technical Document Center
	Commander, U.S. Army Tank-Automotive Command, Warren, MI 48397-5000
1	ATTN: AMSTA-ZSK
2	AMSTA-TSL, Technical Library
	Commander, White Sands Missile Range, NM 88002
1	ATTN: STEWS-WS-VT
	President, Airborne, Electronics and Special Warfare Board, For Bragg, NC 28307
1	ATTN: Library
	Director, U.S. Army Ballistic Research Laboratory, Aberdeen Proving Ground, MD 21005
1	ATTN: SLCBR-TSB-S (STINFO)
	Commander, Dugway Proving Ground, Dugway, UT 84022
1	ATTN: Technical Library, Technical Information Division
	Commander, Harry Diamond Laboratories, 2800 Powder Mill Road, Adelphi, MD 20783
1	ATTN: Technical Information Office
	Director, Benet Weapons Laboratory, LCWSL, USA AMCCOM, Watervliet, NY 12189
1	ATTN: AMSMC-LCB-TL
1	AMSMC-LCB-R
1	AMSMC-LCB-RM
1	AMSMC-LCB-RP
	Commander, U.S. Army Foreign Science and Technology Center, 220 7th Street, N.E., Charlottesville, VA 22901-5396
3	ATTN: AIFRTC, Applied Technologies Branch, Gerald Schlesinger

No. of Copies	To
	Commander, U.S. Army Aeromedical Research Unit, P.O. Box 577, Fort Rucker, AL 36360
1	ATTN: Technical Library
	Commander, U.S. Army Aviation Systems Command, Aviation Research and Technology Activity, Aviation Applied Technology Directorate, Fort Eustis, VA 23604-5577
1	ATTN: SAVDL-E-MOS
	U.S. Army Aviation Training Library, Fort Rucker, AL 36360
1	ATTN: Building 5906-5907
	Commander, U.S. Army Agency for Aviation Safety, Fort Rucker, AL 36362
1	ATTN: Technical Library
	Commander, USACDC Air Defense Agency, Fort Bliss, TX 79916
1	ATTN: Technical Library
	Commander, U.S. Army Engineer School, Fort Belvoir, VA 22060
1	ATTN: Library
	Commander, U.S. Army Engineer Waterways Experiment Station, P. O. Box 631, Vicksburg, MS 39180
1	ATTN: Research Center Library
	Commandant, U.S. Army Quartermaster School, Fort Lee, VA 23801
1	ATTN: Quartermaster School Library
	Naval Research Laboratory, Washington, DC 20375
1	ATTN: Code 5830
2	Dr. G. R. Yoder - Code 6384
	Chief of Naval Research, Arlington, VA 22217
1	ATTN: Code 471
1	Edward J. Morrissey, WRDC/MLTE, Wright-Patterson Air Force, Base, OH 45433-6523
	Commander, U.S. Air Force Wright Research & Development Center, Wright-Patterson Air Force Base, OH 45433-6523
1	ATTN: WRDC/MLC
1	WRDC/MLLP, M. Forney, Jr.
1	WRDC/MLBC, Mr. Stanley Schulman
	NASA - Marshall Space Flight Center, MSFC, AL 35812
1	ATTN: Mr. Paul Schuerer/EH01
	U.S. Department of Commerce, National Institute of Standards and Technology, Gaithersburg, MD 20899
1	ATTN: Stephen M. Hsu, Chief, Ceramics Division, Institute for Materials Science and Engineering
1	Committee on Marine Structures, Marine Board, National Research Council, 2101 Constitution Ave., N.W., Washington, DC 20418
1	Librarian, Materials Sciences Corporation, 930 Harvest Drive, Suite 300, Blue Bell, PA 19422
1	The Charles Stark Draper Laboratory, 68 Albany Street, Cambridge, MA 02139
	Wyman-Gordon Company, Worcester, MA 01601
1	ATTN: Technical Library
	Lockheed-Georgia Company, 86 South Cobb Drive, Marietta, GA 30063
1	ATTN: Materials and Processes Engineering Dept. 71-11, Zone 54
	General Dynamics, Convair Aerospace Division, P.O. Box 748, Fort Worth, TX 76101
1	ATTN: Mfg. Engineering Technical Library
	Director, U.S. Army Materials Technology Laboratory, Watertown, MA 02172-0001
2	ATTN: SLCMT-TML
2	Authors

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
HOT ISOSTATIC PRESSING OF SUPERCONDUCTING CERAMICS -
Kerry T. Richards and CPT Richard H. Benfer

AD______________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Technical Report MTL TR 90-51, October 1990, 19 pp-illus-tables,

Key Words

Ceramic materials
Hot isostatic pressing (HIP)
Superconducting materials

Hot isostatic pressing (HIP) was studied as a method for processing bulk superconductors. Superconducting powder was derived from the calcination of nitrated Y_2O_3, CuO and $BaCO_3$ powder. The powder was HIPed using pressures of 70, 140, and 210 MPa with temperatures of 820°C and 950°C for a hold time of 1 hour. The density, hardness and Young's modulus of HIPed samples were higher than those of sintered control samples. Superconducting transition temperatures >92°K were achieved without post-HIP annealing of the samples.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
HOT ISOSTATIC PRESSING OF SUPERCONDUCTING CERAMICS -
Kerry T. Richards and CPT Richard H. Benfer

AD______________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Technical Report MTL TR 90-51, October 1990, 19 pp-illus-tables,

Key Words

Ceramic materials
Hot isostatic pressing (HIP)
Superconducting materials

Hot isostatic pressing (HIP) was studied as a method for processing bulk superconductors. Superconducting powder was derived from the calcination of nitrated Y_2O_3, CuO and $BaCO_3$ powder. The powder was HIPed using pressures of 70, 140, and 210 MPa with temperatures of 820°C and 950°C for a hold time of 1 hour. The density, hardness and Young's modulus of HIPed samples were higher than those of sintered control samples. Superconducting transition temperatures >92°K were achieved without post-HIP annealing of the samples.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
HOT ISOSTATIC PRESSING OF SUPERCONDUCTING CERAMICS -
Kerry T. Richards and CPT Richard H. Benfer

AD______________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Technical Report MTL TR 90-51, October 1990, 19 pp-illus-tables,

Key Words

Ceramic materials
Hot isostatic pressing (HIP)
Superconducting materials

Hot isostatic pressing (HIP) was studied as a method for processing bulk superconductors. Superconducting powder was derived from the calcination of nitrated Y_2O_3, CuO and $BaCO_3$ powder. The powder was HIPed using pressures of 70, 140, and 210 MPa with temperatures of 820°C and 950°C for a hold time of 1 hour. The density, hardness and Young's modulus of HIPed samples were higher than those of sintered control samples. Superconducting transition temperatures >92°K were achieved without post-HIP annealing of the samples.

U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001
HOT ISOSTATIC PRESSING OF SUPERCONDUCTING CERAMICS -
Kerry T. Richards and CPT Richard H. Benfer

AD______________
UNCLASSIFIED
UNLIMITED DISTRIBUTION

Technical Report MTL TR 90-51, October 1990, 19 pp-illus-tables,

Key Words

Ceramic materials
Hot isostatic pressing (HIP)
Superconducting materials

Hot isostatic pressing (HIP) was studied as a method for processing bulk superconductors. Superconducting powder was derived from the calcination of nitrated Y_2O_3, CuO and $BaCO_3$ powder. The powder was HIPed using pressures of 70, 140, and 210 MPa with temperatures of 820°C and 950°C for a hold time of 1 hour. The density, hardness and Young's modulus of HIPed samples were higher than those of sintered control samples. Superconducting transition temperatures >92°K were achieved without post-HIP annealing of the samples.