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CHARACTERIZATION OF SINTERING PHENOMENA OF $(Na_{0.5}K_{0.5})NbO_3$

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ABSTRACT

Sintering behavior and microstructure development were studied on undoped and barium doped sodium-potassium niobate of the composition $(Na_{0.5}K_{0.5})NbO_3$. Calcining conditions were optimized. A decrease in density accompanied by large grain growth and an increase in pore size were observed. Examinations by scanning electron microscopy revealed a microstructure consisting of well marked cubic grains. Barium appeared to be a powerful grain growth inhibitor. It is postulated that the low surface energy of the (100) planes of sodium-potassium niobate is responsible for the difficulties in conventional sintering of these materials.

This work was done under the auspices of the U. S. Atomic Energy Commission.

At the time this work was done the writers were, respectively, graduate research assistant and professor of ceramic engineering.

* Now at Technical University of Berlin, Germany.

I. INTRODUCTION

Compounds of the perovskite type structure have received considerable interest in recent years because of their ferroelectric properties. The solid solution system formed between $NaNbO_3$ and $KNbO_3$, despite the large difference in the ionic radii of sodium and potassium, is reported to have low dielectric constants and high electromechanical coupling coefficients. Earlier work by Shirane et al.[1] has shown that most of the phases in this system are ferroelectric at room temperature except those very close to the $NaNbO_3$ side of the phase diagram. Sodium-potassium niobate of the composition $(Ka_{0.5}K_{0.5})NbO_3$ is reported to possess the most desirable ferroelectric properties of this solid solution system.[2,3]

Conventional processing of ceramics of sodium-potassium niobates result in poor structures of the fired bodies; in particular, a residual porosity was reported by L. Egerton et al.[4] Furthermore, difficulties seem to exist in sintering these materials to sufficiently homogeneous dense ceramics possessing an adequate fine grained microstructure. As a result of these problems, most of the ceramic work done in the sodium-potassium niobate system is carried out using hot-pressing techniques. Although capable of supplying quality material, hot-pressing is not economical in high volume production.

The purpose of this study was to investigate and to characterize the behavior of the compound $(Na_{0.5}K_{0.5})NbO_3$ in conventional ceramic processing, to determine, and if possible to eliminate, the factors which are constantly leading to poor results in sintering, and to improve the microstructure of sintered samples. The various sintering mechanisms for

solid state sintering and for sintering in the presence of a liquid phase have been discussed in detail by Kingery and Berg,[5] Kingery,[6] Coble[7] and Cahn et al.[8]

II. EXPERIMENTAL PROCEDURES

(1) Powder Preparation

A standard technique was developed and employed for preparation of all powder samples made using carbonates of sodium and potassium. After drying the components at 110°C for 50 h to remove absorbed water, the reagent grade sodium carbonate* and potassium carbonate,** together with niobium pentoxide,† were weighed in proper portions to give the composition $(Na_{0.5}K_{0.5})NbO_3$. The batch was mixed for 50 h in isopropyl alcohol using a rubber lined ball mill with teflon balls to minimize contamination. Isopropyl alcohol was evaporated under continuous stirring to prevent segregation of the heavier constituents. Final drying conducted at 105°C for 24 h was followed by a dry mixing process for 50 h in a "Sweco" vibratory mill using a polyurethane lined container with teflon balls.

Initial calcining experiments were made at 900°C for 6 h in air. X-ray powder diffraction patterns made on -115 mesh portion of the calcined sample revealed a loss in sodium and potassium during calcination. The stoichiometry was corrected on basis of the X-ray intensity of the extra lines observed in the powder pattern. An excess of 1.5 mole % of

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* J. T. Baker Chemical Co., Phillipsburg, N.J.; principal impurities were SiO_2, 0.002%; Ca + Mg, 0.004%; NH_4OH, 0.003%.

** Mallinckrodt Chemical Works, St. Louis, N.Y., principal impurities were PO_4^{---}, 0.001%; Ca + Mg, 0.01%; NH_4OH, 0.01%.

† Alfa Inorganic, principal impurities reported as oxides by American Spectrographic Laboratories Inc., were Al, 0.08%; Si, 0.01%; Cu <0.02%.

both sodium and potassium carbonate was found to be sufficient to eliminate the extra lines in the X-ray powder diffraction patterns.

Powders were prepared with nitrates of sodium and potassium as starting materials for two reasons:

(1) Because of their low decomposition temperatures; and

(2) Because of their high solubility in hot and cold water.

Proper ratios of reagent grade sodium* and potassium nitrate,** were dissolved in distilled and degased water to give the composition $(Na_{0.5}K_{0.5})NbO_3$ when Nb_2O_5 was added. The solution was kept at a temperature of about 90°C and stirred for 2 h. The temperature was then raised and water was inspissated until the stirrer stopped moving due to the high viscosity. Final drying was conducted in an electric oven for 24 h at 110°C. Small portions of the material were calcined for 6 h at 900°C in air. An examination of the stoichiometry showed a loss of sodium and potassium similar to the carbonate prepared materials. An excess of 1.3 mole % of both sodium and potassium nitrate was found to provide stoichiometric $(Na_{0.5}K_{0.5})NbO_3$ after calcination.

(2) Calcination of the Mixed Raw Materials

An attempt to find some useful data in the literature regarding calcining time and temperature failed because of the very widespread of values reported.[9] Calcining temperatures ranged from 750 to 1000°C and calcining times from 1 to 20 h. Therefore, experiments were made to

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* J. T. Baker Chemical Co., Phillipsburgh, N.J.; principal impurities were: Cl, 0.001%; R_2O_3, 0.002%.

** Principal impurities were: Cl, 0.0027; R_2O_3, 0.0005%.

determine and optimize both calcining time and temperature using the density obtained in a standard sintering treatment (1050°C for 2 h in air) as a guide. The optimal conditions for calcining in air of sodium-potassium niobate of the composition $(Na_{0.5}K_{0.5})NbO_3$ prepared with carbonates were determined to be 850°C and 2 h. Some materials calcined at other than the optimal conditions showed a drop in sintered density of 8% or more. The assumption was made that the same conditions would be valid for calcining sodium-potassium niobate powders prepared with nitrates as starting materials. Final particle size measurements employing a scanning electron microscope showed a particle size distribution range between 0.2 and 1.5 μm. Figure 1 shows a SEM micrograph of a representative portion of calcined powders after milling in a polyurethane lined container with teflon balls for 24 h. After all processing steps, the sodium-potassium niobate powders were analyzed by semi-quantitative analysis.*

(3) Sintering

Pellets of 3/4 inch in diameter were cold pressed at 20,000 psi using a tungsten alloy steel die. The pellets ranged in weight from 3 to 4 grams. In order to improve the green strength of the pellets, the powder to be pressed was wetting slightly by adding approximately 2 cm^3 isopropyl alcohol to 100 gr of dry powder. Green densities obtained ranged from 61 to 64% of theoretical depending upon the composition of the batch. The pressing process was followed by a drying step at 105°C for 24 h to evaporate the added alcohol.

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* American Spectrographic Laboratories, Inc., principal impurities were Al, 0.12%; Fe, <0.01%; Ca, 0.02% reported as oxides in weight percent.

Preliminary determinations of the influence of sintering atmospheres on density and weight loss suggested firing in a flowing oxygen gas atmosphere. Because of the high vapor pressures of the alkali oxides, it was necessary to use packing powder techniques to provide a well defined activity of both sodium and potassium through the whole sintering process. Undoped packing powder of the composition $(Na_{0.5}K_{0.5})NbO_3$ having 3 mole % excess in alkali carbonates was used for all sintering experiments.

After loading the furnace, it was evacuated to approximately one-thousandth of an atmosphere and refilled with oxygen gas. The heating rate was 15°C per minute. After firing, all sintered specimens were partly quenched in air by removing the crucible from the furnace. As the pellets reached a temperature of about 400°C they were put into a dessicator for further cooling to room temperature. This precaution was necessary because of the hygroscopic nature of some specimens, especially of pellets sintered for short periods of time and of pellets having low densities.

Measurements of the sintered density were made using a mercury immersion apparatus. Specimens used for density measurements were carefully checked for small cracks. The accuracy of these measurements was approximately ± 0.5%.

III. RESULTS AND DISCUSSION

(1) Sintering Behavior and Density

The relative density of undoped carbonate prepared samples sintered for varying times at 1000, 1050 and 1100°C in oxygen atmosphere are given in Fig. 2. The initial densification rate shows a rapid increase

with increasing remperature. This increase may be due to an increasing amount of liquid phase present as the sintering temperature is increased, accompanied by a higher diffusivity of Nb^{5+}, which was thought to be the sintering rate determining species because of its very high ionic potential and its low vapor pressure. Samples sintered at 1000 and 1050°C show the typical sintering behavior for sintering in presence of a liquid phase, whereas the sample sintered at a temperature of 1100°C with its very high initial densification rate shows a decrease in density after a transition stage. This de-densification is not accompanied by a large weight change of the samples as can be seen from Fig. 3.

Figures 4, 5, and 6 show the relative density of 0.5, 1.0 and 4.0 atomic % barium doped carbonate prepared samples sintered for varying times at 1000, 1050 and 1100°C in oxygen atmosphere. The initial densification rate undergoes a maximum in the range of 0.5 atomic % dopant and indicates a considerable amount of liquid phase present. Furthermore, the de-densification effect seems to have a maximum value in the same region. The weight changes versus linear sintering time for samples sintered at 1100°C, as given in Fig. 3, indicate a maximum in weight loss for the 0.5 atomic % barium doped material. The material doped with 1.0 atomic % barium shows an initial weight gain followed by a weight loss if sintering continues. For this material, weight gain and de-densification coincides. Samples doped with more than 2.0 atomic % barium show a constant weight gain during the sintering process which probably indicates the development of an increasing amount of a second phase on the surface of the grains. No de-densification could be observed in the highly doped material. The initial densification rate

drops noticeably with increasing doping level. An examination of the microstructure of samples doped with varying amounts of barium employing a SEM showed a reduction of grain size by a ratio of approximately 25 to 1 caused by raising the doping level from 0 to 4.0 atomic %.

Small amounts of barium doped into the material seem to decrease the activities of sodium and potassium in the samples so that the pellets start losing weight, whereas higher amounts of barium lead to an increasing development of a barium rich second phase on the surface of the grains, which will be discussed later.

Figure 7 shows the relative density of 0.5, 1.0 and 4.0 atomic % barium doped nitrate prepared samples sintered for varying times at 1100°C in oxygen atmosphere. The sintering behavior of this material differs noticeably from the sintering behavior of the carbonate prepared material. All curves level after a short sintering time and no further densification is observed. No de-densification effect could be observed. Sintering of pellets prepared with nitrates and of all pellets prepared with carbonates as well, was conducted in packing powder of the composition $(Na_{0.5}K_{0.5})NbO_3$ having high activities of sodium and potassium.

Further sintering experiments employing the same nitrate prepared materials were carried out using packing powder with an excess of Nb_2O_5, i.e., a lower activity of sodium and potassium. These results are given in Fig. 8. The sintering characteristics of these specimens indicate a lower or negligible liquid phase content. Fairly high densities could be obtained with the 0.5 atomic % barium doped material after a sintering time of 15 h at 1100°C. No de-densification effect could be observed. Because of the low liquid phase content due to the change in the packing

powder composition, it would be possible to raise the sintering temperature and to achieve high densities in much shorter times.

(2) Pore Growth and De-Densification

The accelerated densification rate due to the presence of a liquid phase brings the samples to high densities in a very short time where a large portion of the pores can be assumed to be closed. Usually, there will be a negative pressure in each pore given by the relation

$$P = \frac{-2\gamma_{LV}}{r_p} \qquad (1)$$

P = Pressure

γ_{LV} = Surface energy (liquid vapor)

r_p = pore radius

However, if a gas is trapped inside the pores, diffusion of the gas to the surface may become negligible compared with the diffusion flux between the pores and a volume expansion may occur. Gupta and Coble[10] presume that the driving force for densification may approach zero or become negative when the gas trapped inside the pores equilibrates with the surface tension and the gas is relatively insoluble in the matrix material. In this case, one or more of the pore growth mechanisms, pore-pore material transfer or pore migration, could become effective. In general, however, a decrease in density implies either a weight loss or a volume expansion or both.

The constant weight of the undoped carbonate prepared samples during de-densification indicates an equilibrium situation between the activities of sodium and potassium in the sample and packing powder. Therefore, the

material leaving or entering the sample during de-densification is negligible. Thus, the de-densification has to occur by volume expansion exclusively.

The series of SEM micrographs in Fig. 9 are of undoped samples sintered at 1100°C for varying times; their densities are shown in Fig. 2. Figure 9A shows a sample sintered for 15 min. The microstructure of the fired body shows a relatively uniform grain size with a few large grains interspersed. All grains show a well-developed cubic shape. Figure 9B taken after a sintering time of 1 h shows a very inhomogeneous microstructure with a few grains having a size of approximately 15 to 20 μm and a large number of smaller grains having a size of 1 to 3 μm. Figure 9C taken on the same material after a sintering time of 4 h, reveals that all grains with a diameter less than 5 μm have disappeared, leaving a more open structure. A comparison with Fig. 2 shows that this sample has exhibited a considerable de-densification. Figure 9D shows a sample after a sintering time of 8 h. This material exhibits a very open microstructure with large grains and open pores.

These figures suggest that the material originating from the small grains is transferred to the large grains which are growing. Because the de-densification during grain growth is not accompanied by a weight change, the centers of the large grains have to move apart. This possibility is given by assuming a liquid layer between the cubic grains as proposed in the model of sintering in presence of a liquid phase. This assumption is valid because the two-phase diagrams of the system Na_2O-Nb_2O_5 and K_2O-Nb_2O_5 indicate a liquid phase in this temperature region (1100°C) if excess Na_2O and K_2O are present.

If the initial density is sufficiently high to provide a large portion of closed pores, small amounts of CO and CO_2 gas generating from a late decomposition of $BaCO_3$ with its very high decomposition temperature of 1450°C may equilibrate with the surface tension and stop or reverse densification. The de-densification effect appears more rapid by the introduction of $BaCO_3$ but disappears if the amount of $BaCO_3$ exceeds 2 mole %. The disappearance of the de-densification effect is accompanied by a noticeable decrease in the initial densification rate. Furthermore, it is interesting that all samples exhibiting de-densification attain a final density of approximately 80% of theoretical.

The coincidence of a decreasing initial densification rate with the disappearance of de-densification despite an increasing amount of $BaCO_3$ introduced, indicates that the amounts of CO_2 and CO gas are sufficiently high that the gases can leave the sample without allowing large portions of closed pores to be formed. The remaining barium carbonate or barium oxide may obtain sodium and potassium from the packing powder permeating through these open pores to form the barium-rich second phase. This mechanism would also account for the decreasing initial densification with increasing $BaCO_3$ content and for the steady weight gain even after relatively long sintering times. The important role of CO_2 and CO gases, which can be assumed to be quite insoluble in the matrix material, in de-densification of sodium-potassium niobate is supported by the fact that this effect could not be observed in any experiment carried out with nitrate prepared materials.

(3) Surface Energy and Driving Force for Sintering

The driving force for every sintering mechanism proposed is the decrease of the surface free energy due to the decrease of surface area during densification. Assuming a complete wetting of the solid phase by the liquid phase and a penetration between grains in a liquid phase sintering model, the following surface energy relationships are required:

$$\gamma_{sv} > \gamma_{lv} > \gamma_{ss} > 2\gamma_{sl} \qquad (2)$$

where s, l, and v refer to solid, liquid and vapor respectively.

In the process with appreciable liquid present or at least sufficient liquid to cover all solid surfaces, the driving force for densification becomes the liquid-vapor surface energy. Even though liquid-vapor surface energies are lower than solid-vapor surface energies, the increase in diffusivity in liquids relative to solids greatly enhances initial densification rates. In undoped sodium-potassium niobate the high initial densification is accompanied by a rapid grain growth leading to an equilibrium microstructure. The microstructure of all samples sintered in packing powder with high activities in sodium and potassium consisted of cubic grains. Figure 10A shows a SEM micrograph of the equilibrium microstructure of a 0.5 atomic % barium doped nitrate prepared sample sintered for 8 h at 1100°C in oxygen atmosphere. Figure 10B shows the same nitrate prepared material sintered at 1100°C for 15 h in packing powder of the same composition but with added Nb_2O_5 to give low activities of sodium and potassium.

The great difference between the microstructures shown in these two figures was achieved only by a slight change of the packing powder from the sodium and potassium-rich to the niobium-rich side of the compound $(Na_{0.5}K_{0.5})NbO_3$. The sample in Fig. 10A shows an equilibrium microstructure where the driving force for a further densification has become extremely low, whereas Fig. 10B shows a sample having a density of 96% of theoretical (4.51 g/cm^3). The grains in this sample are quite irregular and show no distinct planes.

In Fig. 10A all grains are cubes having very sharp edges and distinct corners. Intersections of grains are very sharp; no necks can be observed. All crystallographic planes obtained in the microstructure of all samples sintered in packing powder having high activities in sodium and potassium are believed to belong to the cubic {100} family.

It is obvious that this plane must have the lowest surface energy of all possible planes. This becomes more clear if one considers the perovskite type unit cell of $(Na_{0.5}K_{0.5})NbO_3$. The {100} planes consist only of the large oxygen ions and sodium and potassium ions which have low ionic potentials. This configuration resembles the configuration of the {100} planes in NaCl which are known for their low surface energy. Every other plane in the unit cell of sodium-potassium niobate would expose a niobium (5+) to the surface which would imply a drastic increase of the surface energy. The low surface energy of the {100} planes in contrast with the much higher surface energies of all other possible planes is probably the reason for the absence of any necks in the microstructure and the development of very distinct edges and corners of the cubic grains. Furthermore, this low surface energy of the cubic planes

is probably the reason for the reduced sintering rate due to the reduction of the driving force for densification, γ, as proposed in all sintering models.

IV. SUMMARY

An investigation of the sintering behavior of barium doped and undoped sodium-potassium niobate of the composition $(Na_{0.5}K_{0.5})NbO_3$ was conducted.

(1) The optimal calcining conditions with respect to the sintered density of $(Na_{0.5}K_{0.5})NbO_3$ prepared with carbonates were found to be 850°C and 2.0 h.

(2) A decrease in density for undoped $(Na_{0.5}K_{0.5})NbO_3$ after a sintering time of 2 h at a temperature of 1100°C was observed. This de-densification effect was accompanied by a disappearance of all smaller grains in the microstructure and an increase of the pore size.

(3) The de-densification was found to be favored by small amounts of barium doped into the material but inhibited by additions of more than 2 atomic % barium. The de-densification has a maximum in the range of 0.5 atomic % barium.

(4) It was found that de-densification in undoped sodium-potassium niobate occurs without any detectable change in weight of the samples, which implies a negligible amount of material is entering or leaving the sample during firing. Furthermore, it implies an equilibrium between the activities of all elements in sample and packing powder.

(5) Barium (2+) was found to inhibit grain growth. Even after sintering times of 8 h at a temperature of 1100°C the microstructure in 4.0 atomic % doped material remained very uniform and the grain size was

less than 1 μm.

(6) A slight change in composition of the packing powder from the sodium and potassium-rich side of $(Na_{0.5}K_{0.5})NbO_3$ to the niobium-rich side was found to have a large influence on sintering behavior and density, by changing the sintering mechanism from liquid phase to solid state sintering.

(7) Indications were found that additions of more than 2.0 atomic % barium lead to a formation of a barium-rich second phase on the surface of the grains. It is possible that this second phase is mainly responsible for the decrease in grain size.

(8) It is postulated that the low surface energy of the {100} planes of $(Na_{0.5}K_{0.5})NbO_3$ formed is the main reason that during conventional sintering the driving force for densification is lowered and densification stops.

ACKNOWLEDGMENT

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FIGURE CAPTIONS

Fig. 1. Scanning electron micrograph of $(Na_{0.5}K_{0.5})NbO_3$ powder prepared with nitrates.

Fig. 2. Relative density of undoped sodium-potassium niobate versus sintering time.

Fig. 3. Weight changes of barium-doped and undoped sodium-potassium niobate prepared with carbonates.

Fig. 4. Relative density of 0.5 atomic % barium doped sodium-potassium niobate versus sintering tiem.

Fig. 5. Relative density of 1.0 atomic % barium doped sodium-potassium niobate versus sintering time.

Fig. 6. Relative density of 4.0 atomic % barium doped sodium-potassium niobate versus sintering time.

Fig. 7. Relative density versus sintering time of barium doped sodium-potassium niobates prepared with nitrates and sintered in packing powder with excess sodium and potassium.

Fig. 8. Relative density versus sintering time of barium doped sodium-potassium niobate prepared with nitrates and sintered in packing powder with excess niobium.

Fig. 9. Scanning electron micrograph showing grain growth behavior of undoped sodium-potassium niobate sintered at 1100°C for A. 15 min; B. 1 hr; C. 4 hrs; D. 8 hrs.

Fig. 10. Scanning electron micrograph showing nitrate prepared material doped with 0.5 atomic % barium sintered at 1100°C in

A. Packing powder with high activities in sodium and potassium;

B. Packing powder with low activities in sodium and potassium.

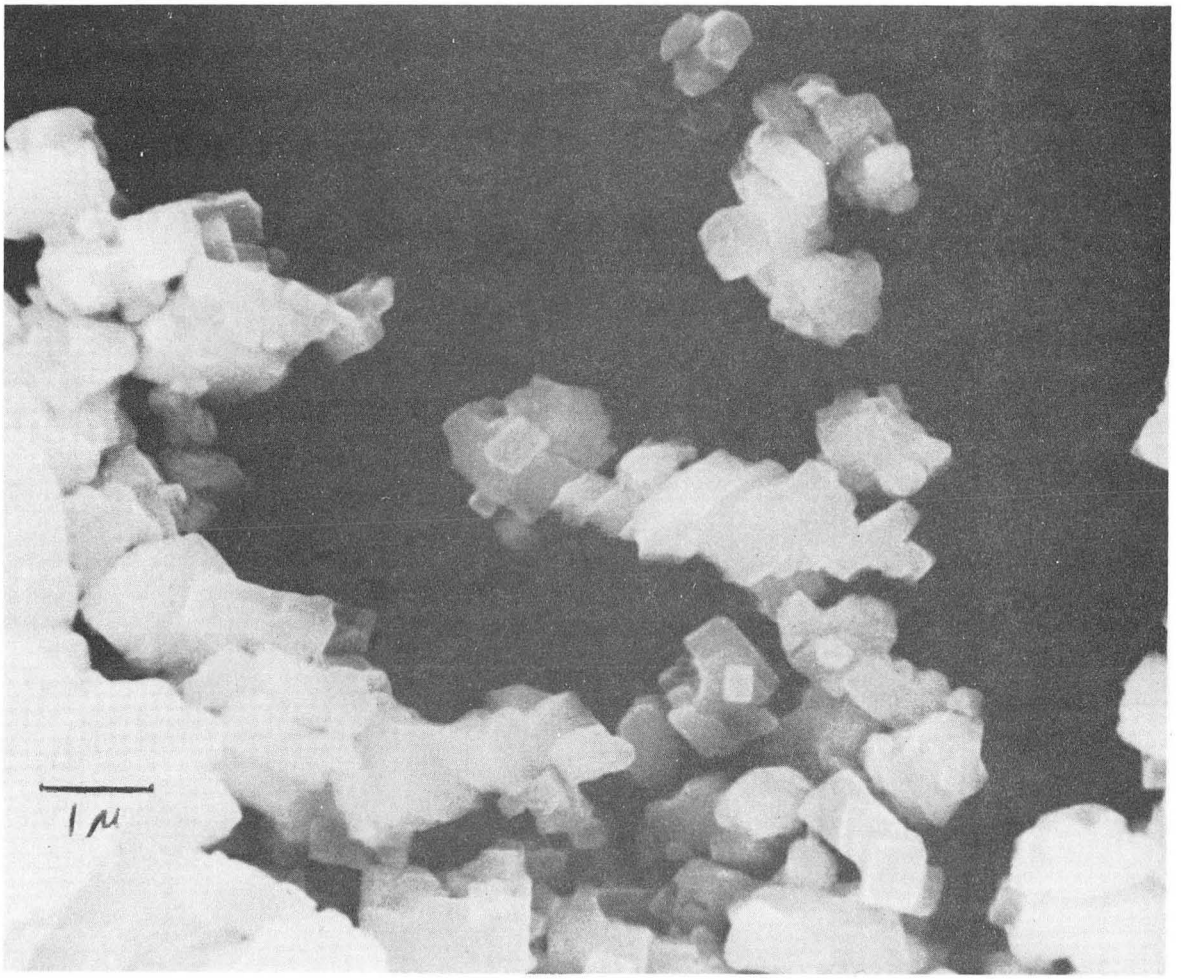

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Fig. 1.

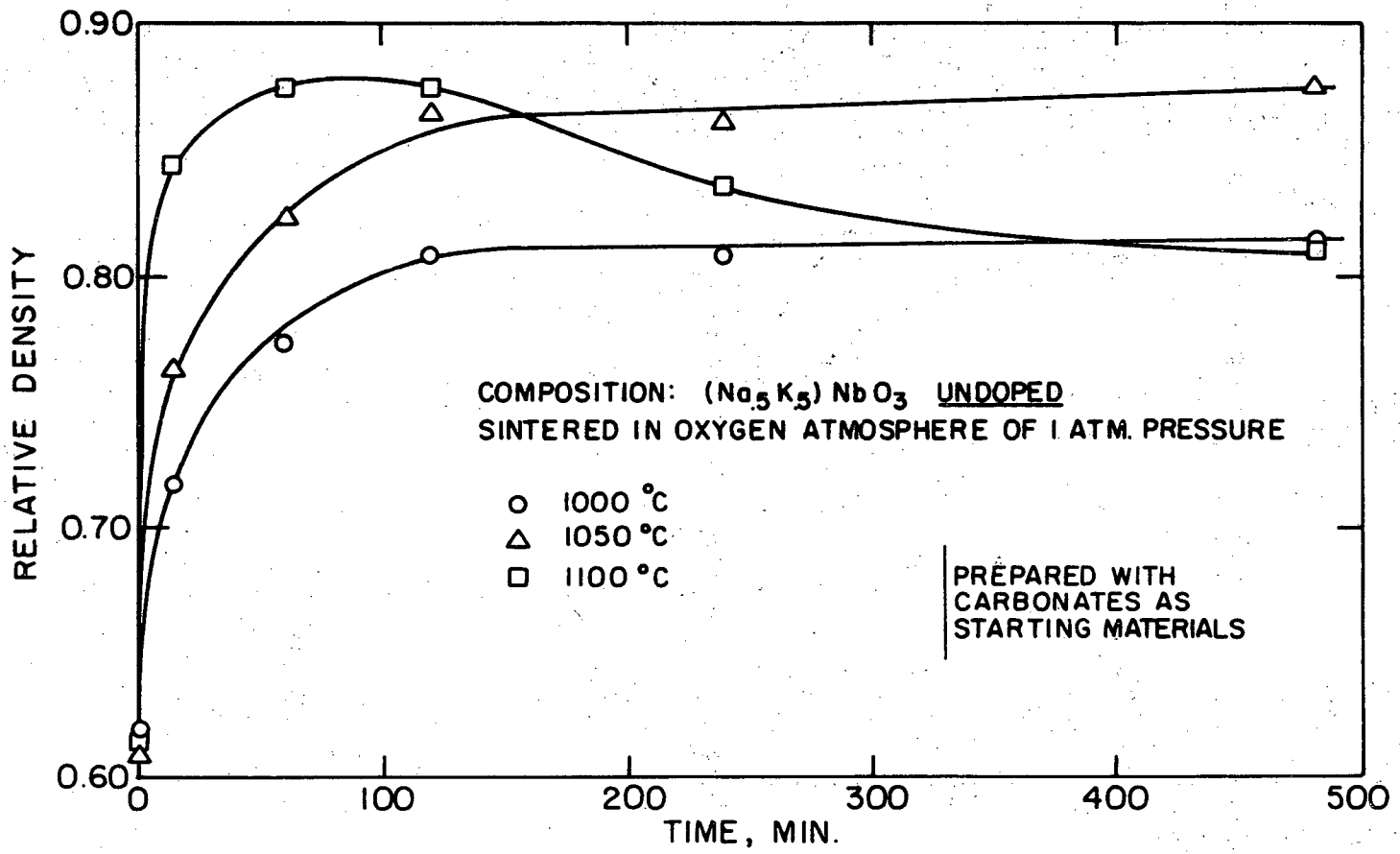

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Fig. 2.

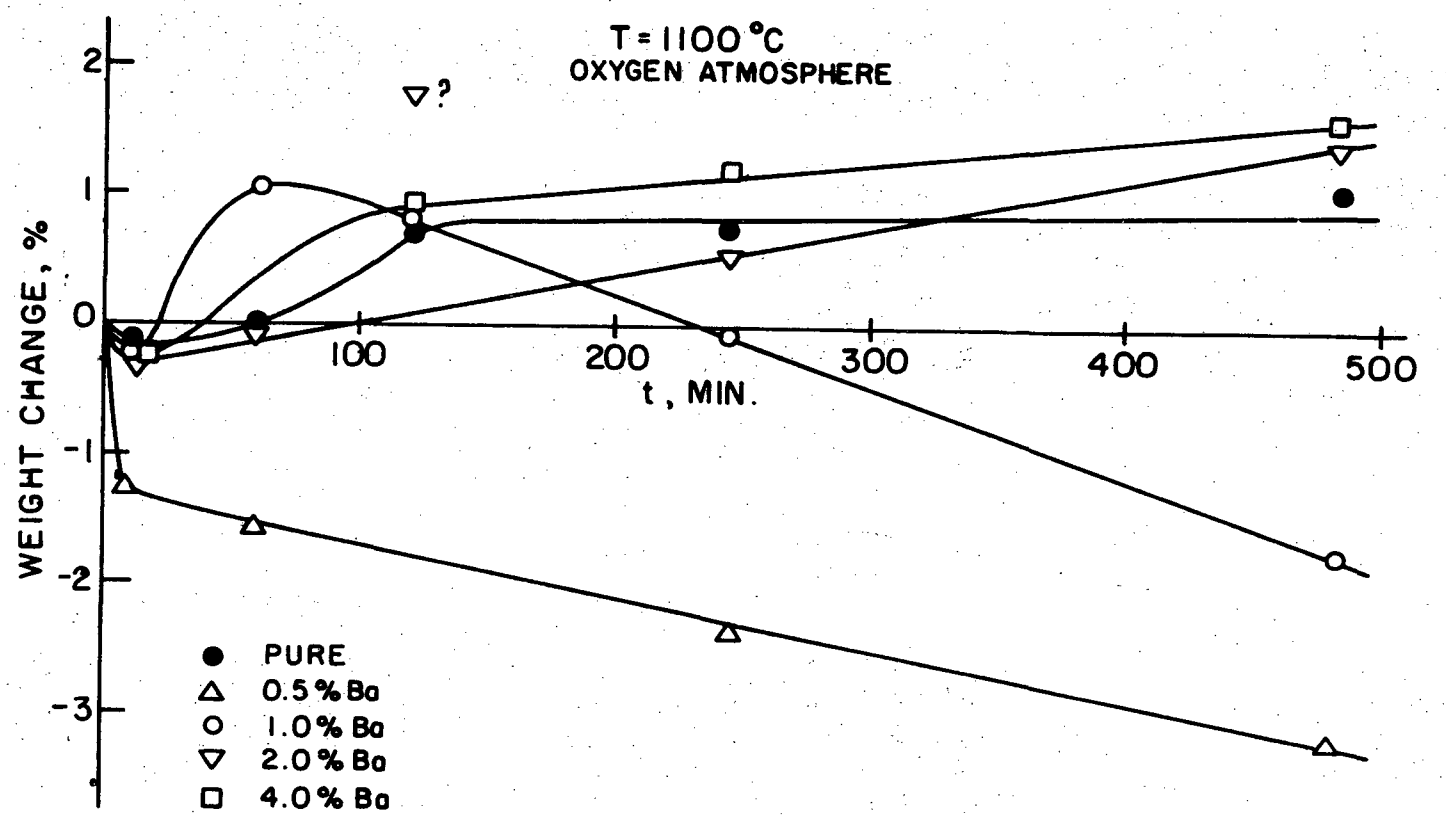

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Fig. 3.

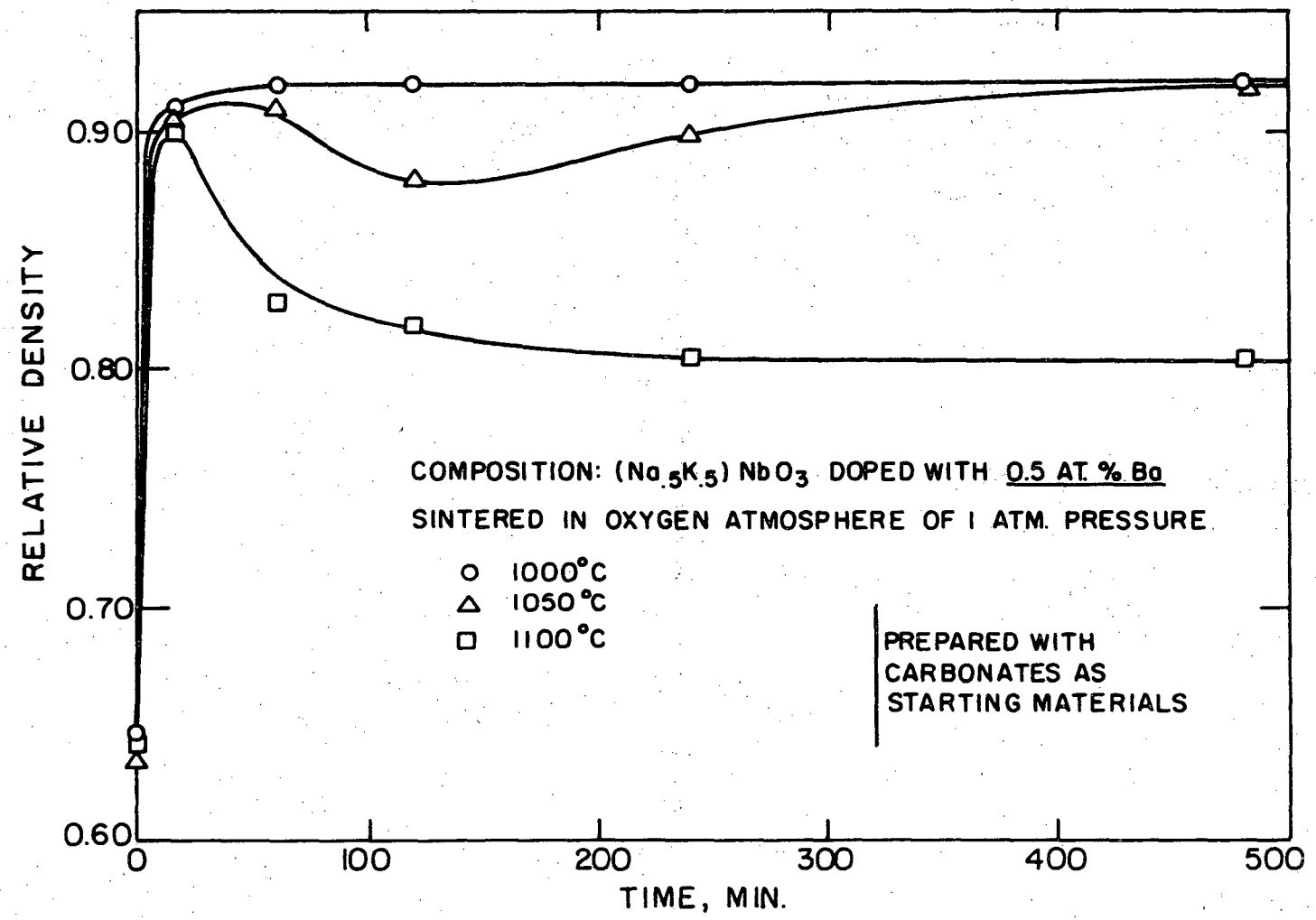

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Fig. 4.

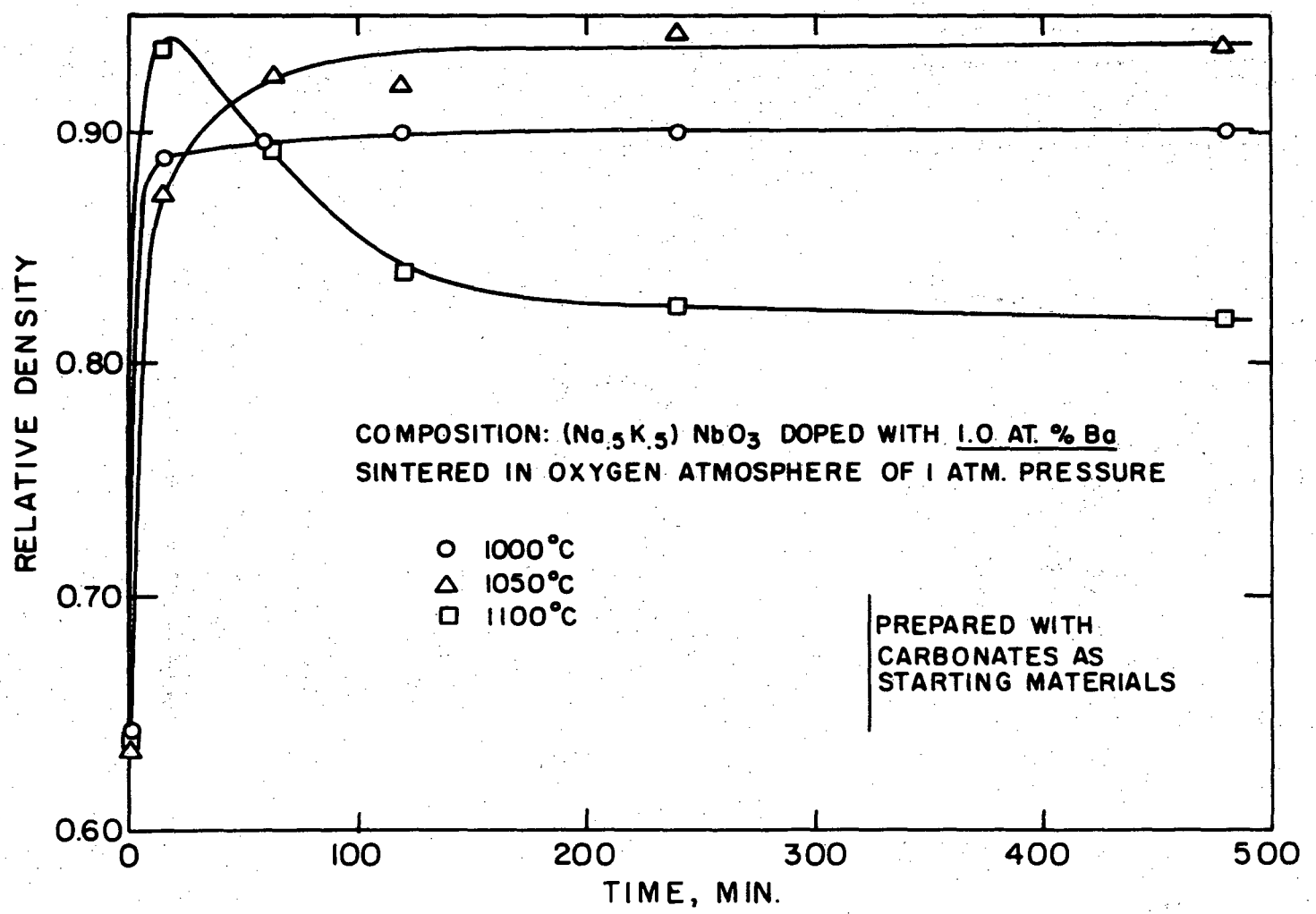

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Fig. 5.

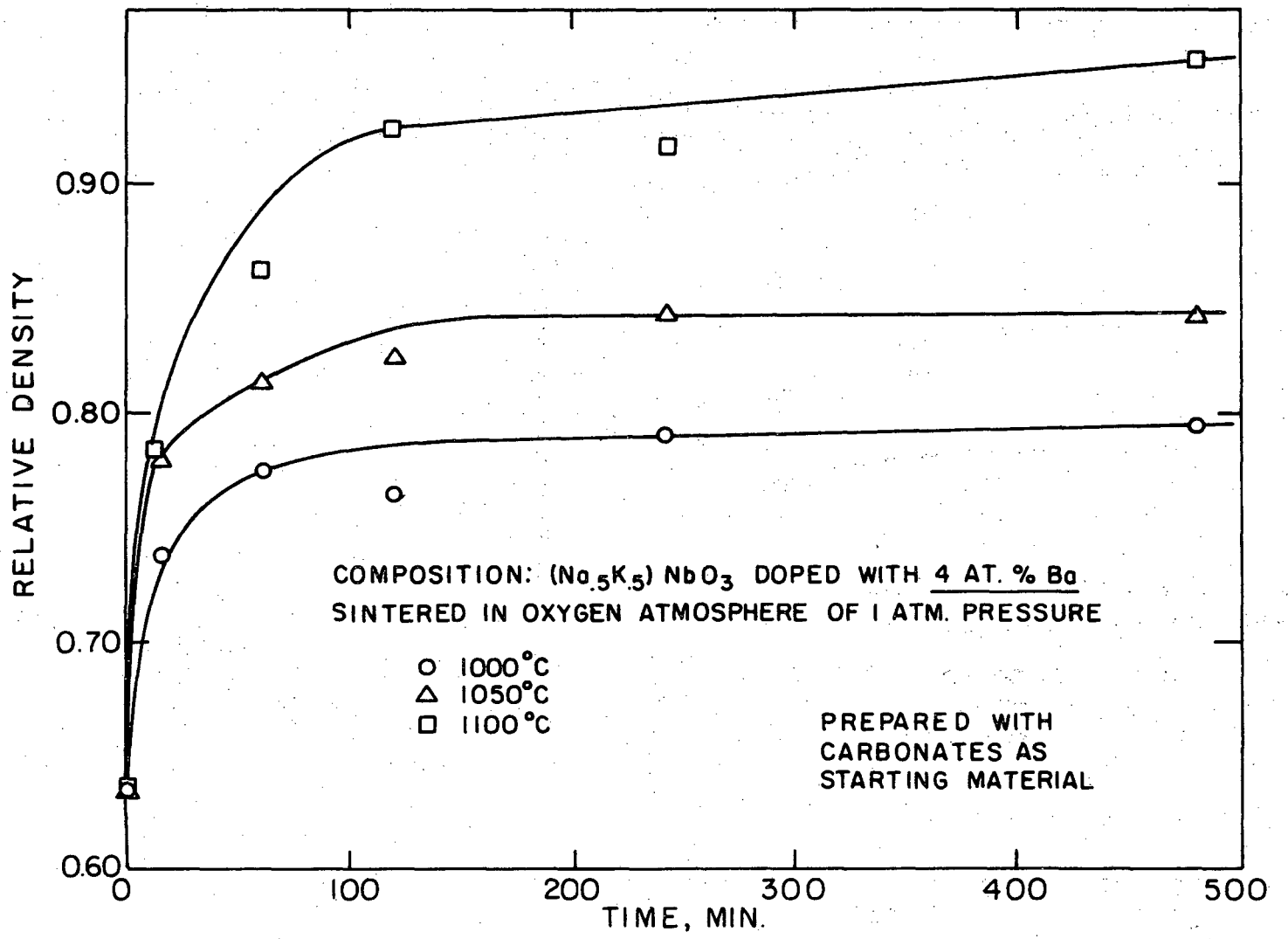

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Fig. 6.

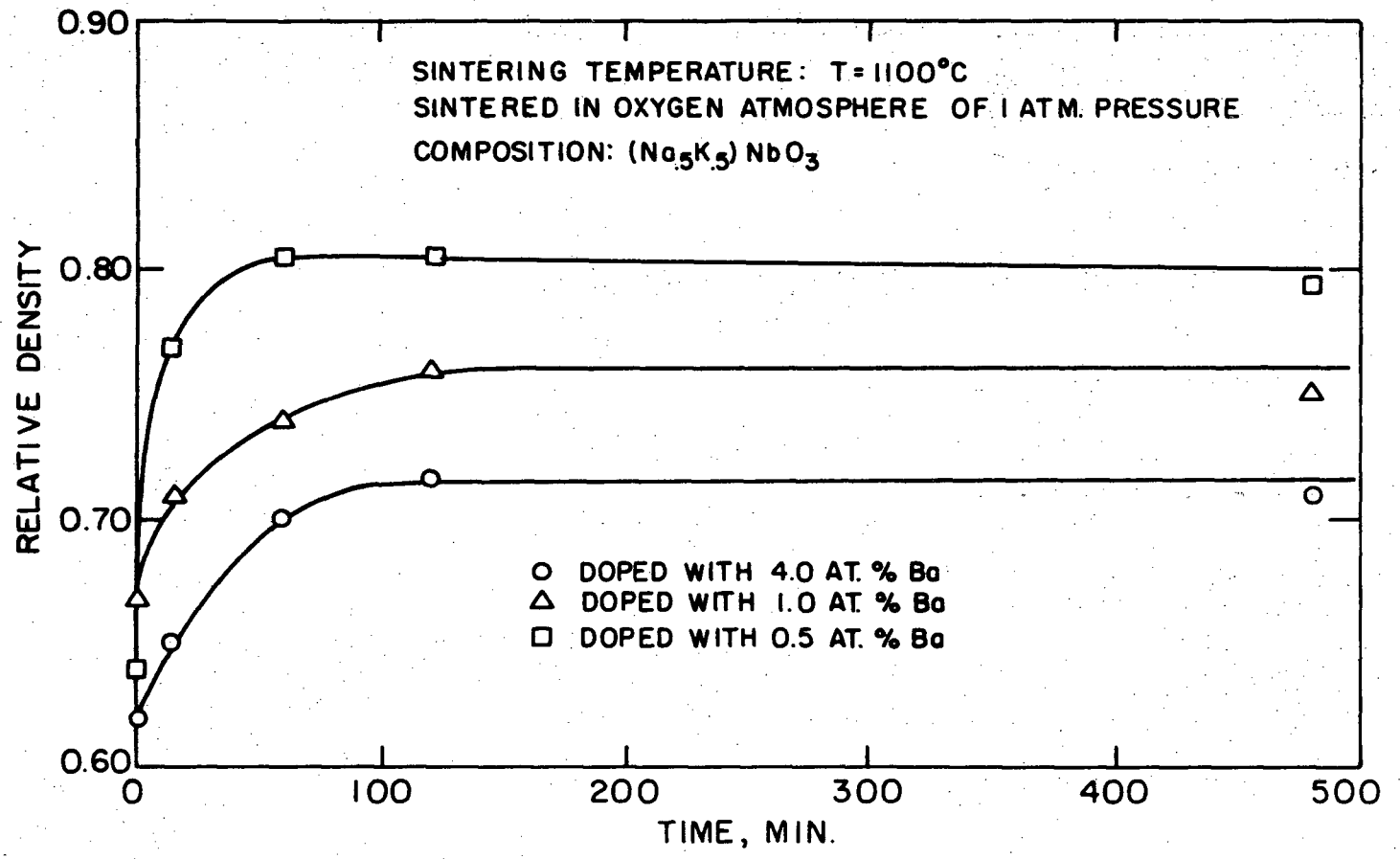

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Fig. 7.

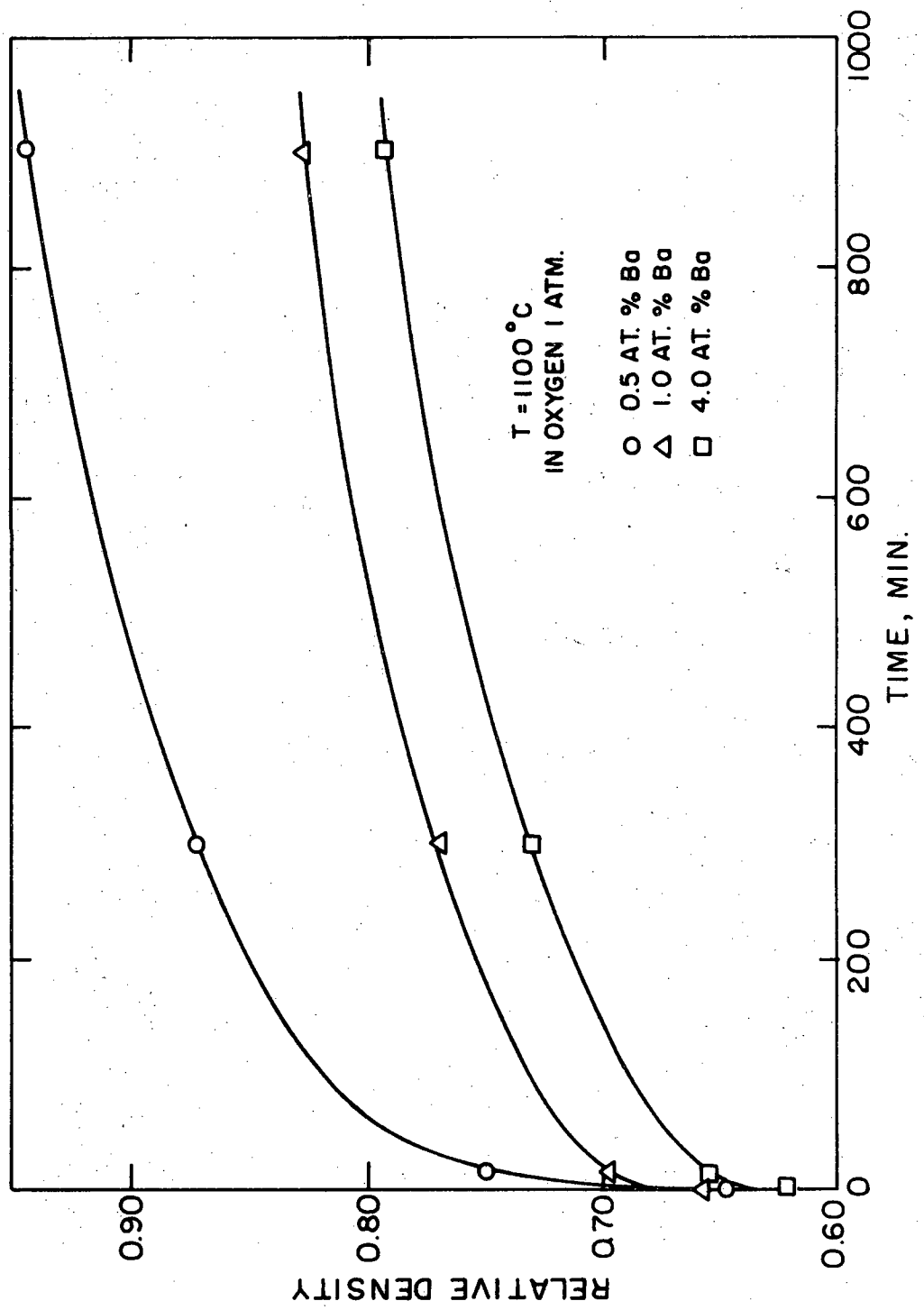

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Fig. 8.

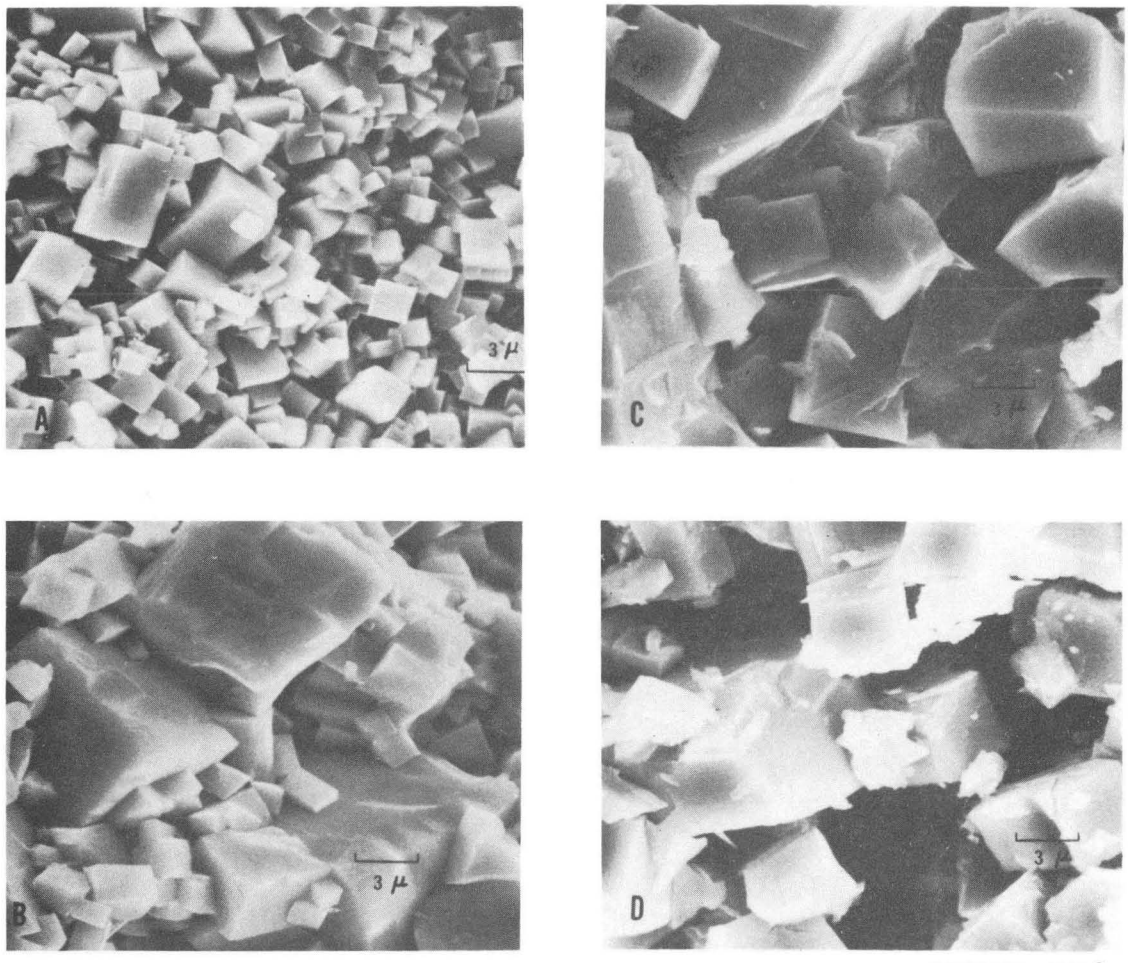

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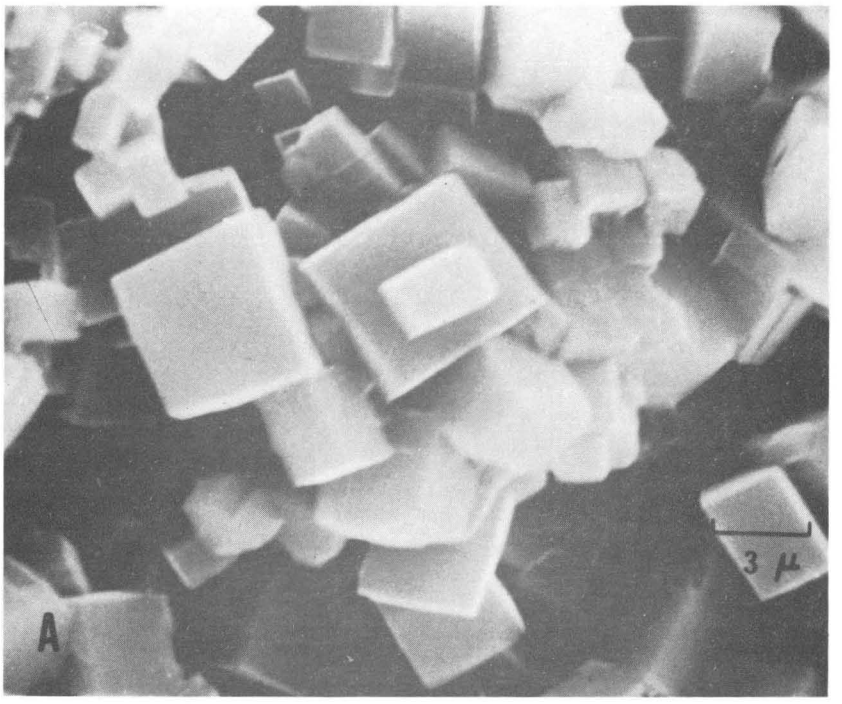
Fig. 9.



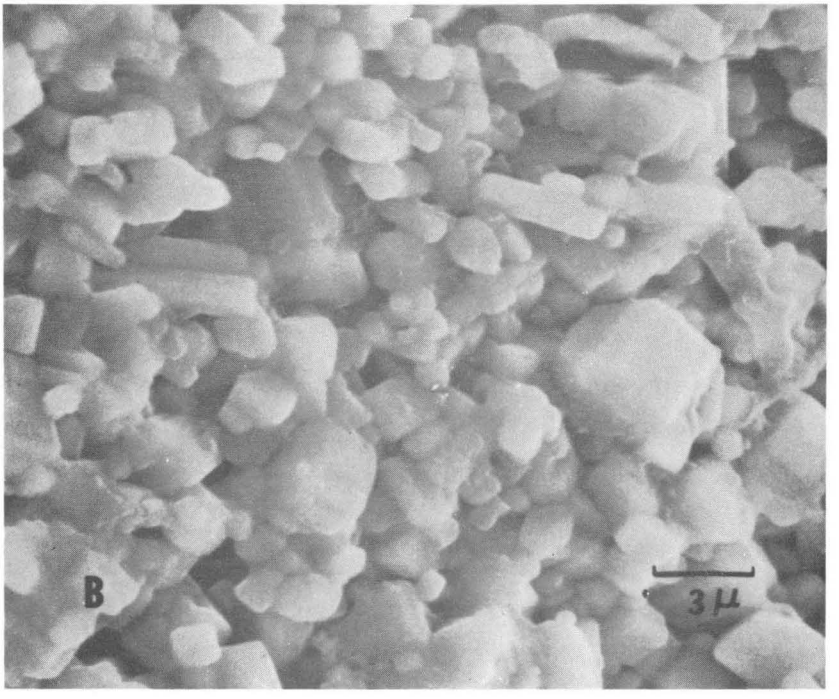


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Fig. 10.

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