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Roll Forming Strip From Oxide Powders

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By Henry M. Harris, Bob L. Forkner, and Hal J. Kelly

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ROLL FORMING STRIP FROM OXIDE POWDERS

by

Henry M. Harris,[1] Bob L. Forkner,[2] and Hal J. Kelly[3]

ABSTRACT

The roll forming of oxide powders was studied in a Bureau of Mines investigation to find the conditions and parameters required to make satisfactory ceramic strip. High-quality porous or dense alumina, zirconia, and porcelain shapes were made by sintering strip roll-formed at room temperature from powders containing minor amounts of binder and water. Strips with thicknesses of 0.04 to 0.08 inch and bulk densities from 40 to 90 percent of theoretical densities were made. The parameters most important to formability were particle size, condition of the powders, and roll gap. Other factors affecting quality were speed and surface condition of the rolls, amount and type of binder, water content, and sintering conditions. Ceramic strip was formed at rates up to 78 feet/minute, which is 13 to 23 times faster than reported by previous investigators, and forming was done in a simple and inexpensive roll unit. Reshaping the strip was possible by cutting, grinding, shearing, and stamping.

The following innovations were developed that allow roll processing not normally possible with ceramic powders: (1) Fine-sized powders were made into strip by preparing these as partially sintered granules; (2) thick strip was formed by pressure feeding powders into the rolls; (3) flexible strip was made using a rubber-base bonding agent; (4) corrugated strip was formed on shaped rolls; and (5) porcelain strip was densified and reduced in thickness by rerolling while hot.

INTRODUCTION

The research reported in this paper is a part of the Bureau of Mines effort to promote the conservation and efficient use of the Nation's mineral resources. Research with the broad aim of developing more effective ceramic processing methods may reduce the fuel consumed in production and the waste of minerals in defective products.

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The roll forming of ceramic strip is a relatively new process. While surveys of ceramic forming (1)[4] and powder metallurgy (14) mention roll forming powders, research was needed to obtain information for developing two other new and related processes--the roll forming of hot powders and the hot rolling of oxide-glass shapes, already reported (5). With this overall objective in view, the work on cold rolling ceramic powder compositions was specifically concerned with identifying the critical parameters in the process. The research did not extend to optimizing the process or products.

A patent by Ragan (15) discloses a method for roll forming ribbon from free-flowing ceramic powders of tile or veneer-type compositions and gives means for shaping, glazing, and mounting the flat shapes made from the ribbon. The forming of oxide and porcelain strip, as in the present work, was not mentioned. Fargo (3) roll-formed urania (UO_2) strip from plasticized powders and sintered, stamped, or cut shapes of strip of high densities. Much of the detail sought on the forming parameters in the present investigation was not given in either of these references. Information was needed on means for avoiding or eliminating problems in forming strip from extremely fine-sized powders and on making thicker strip without changing roll size. Studies also were required to determine if strip could be made at a faster rate. Detailed information was needed on the effect on forming and sintered quality of parameters such as powder-particle size, roll gap, and type and amount of binders. Reports by Ignatiev and coworkers (6) on roll forming a carbide powder and by Sheinberg (17) on rolling Teflon[5] powder have provided some detailed tests results, but these were not published when the present work was started. A flexible rubber binder was found usable by Ignatiev, as in the present work. Uranium dioxide powders sheathed in metal tubes were densified by tandem rolling in a study by Lingafelter (11), but this was for a specific use that permitted the high cost of metal sheathing.

The conditions for forming ceramic powders into green strip are similar to those for metal powders. An exception is that plasticizing binders are required for ceramic powders, which are not as malleable as metal powders. The review by Franssen and Franssen (4) provides insight into the powder rolling of metals and the important conditions and parameters that must also be examined for ceramic powders. Katashinskii and Vinogradov (7) made detailed pressure measurements during the powder rolling of metals, and Vinogradov and Fedorchenko (18) studied the effect of gas and vacuum atmospheres on the powder forming of metals. Neither of these areas has been investigated for ceramic powders.

Work on metal matrix composites containing an oxide by Ready and coworkers (16) and research on bimetal laminates from powders by Katrus and Vinogradov (8) are examples of the versatility of the process for combining dissimilar materials. No similar work on combining different ceramic materials is known. An approach to obtaining thicker plate is given by

[4]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

[5]Reference to specific brands of materials is made for identification only and does not imply endorsement by the Bureau of Mines.

Matsumura and coworkers (12) who rolled iron powders between preformed iron strip. The problem of making thicker strip with a given roll diameter is also found when roll forming ceramic powders.

The roll forming process is essentially simple; it consists of feeding a binder-coated, slightly damp but flowable powder to a set of rotating rolls which compress the powder into a continuous strip. Strip was roll-formed at room temperature with powders containing minor amounts of binder and water; then the dried strip was densified by sintering. Various types of alumina, calcia-stabilized zirconia, and various porcelain, or mixed oxide, compositions were tested. Several samples from each strip composition were sintered at three different temperatures. The compositional parameters investigated that were important to forming strip were the type and particle size of the powders, the type and amount of binder, and the amount of water. The processing conditions studied were powder preparation and mixing methods, effects of equipment variables on forming, such as roll speeds, surfaces, and gaps, and sintering temperatures and conditions. Bulk density, shrinkage, and porosity were measured to evaluate strip formability and quality. Physical property values reported are the arithmetical average for several samples from each type of strip processed at the conditions specified. Dry and sintered bulk densities were the main physical properties used in judging the effect of composition and procedures on formability and quality of the various types of strip made. The quality and formability are compared for strip made from coarse, fine, and granulated powders of the same chemical identity. The relationships of strip composition, powder preparation, and strip formability to dry and sintered bulk densities of the strip are reported in several tables.

Special types of strip were made. Some types, made with rubber binder, were flexible when dry, and one was corrugated rather than flat. A combined process was developed for hot-rolling densification of porcelain strip preformed by powder rolling. The combined procedure parallels those commonly used for making dense metal strip, such as described for nickel by Blore and coworkers (2), for molybdenum and tungsten by Lenz and Peterson (9), for beryllium by Moyer and Sheinhartz (13), and for iron and copper in a book edited by Leszynski (10).

EQUIPMENT AND PROCEDURES

Roll Forming Apparatus

Figure 1 shows the unit used for roll forming powders into strip. The machine consists of a vibratory feeder, feed chute, rolls, offbearing chute, and motor. One roll was mounted on an adjustable slide that allowed change in roll gap and thus in strip thickness. The counterdriven rolls were both powered by a chain-and-sprocket drive from a gearhead motor. A torque-overload device prevented damage to the rolls or drive by jamming or overloading. Roll inserts with a diameter of 5 inches and a width of 2-1/2 inches were used for the majority of tests. The rolls were built small and low to permit later use under furnaces. Even though the rolls did not have some features used or recommended by previous researchers, no major equipment-related processing problems were encountered.

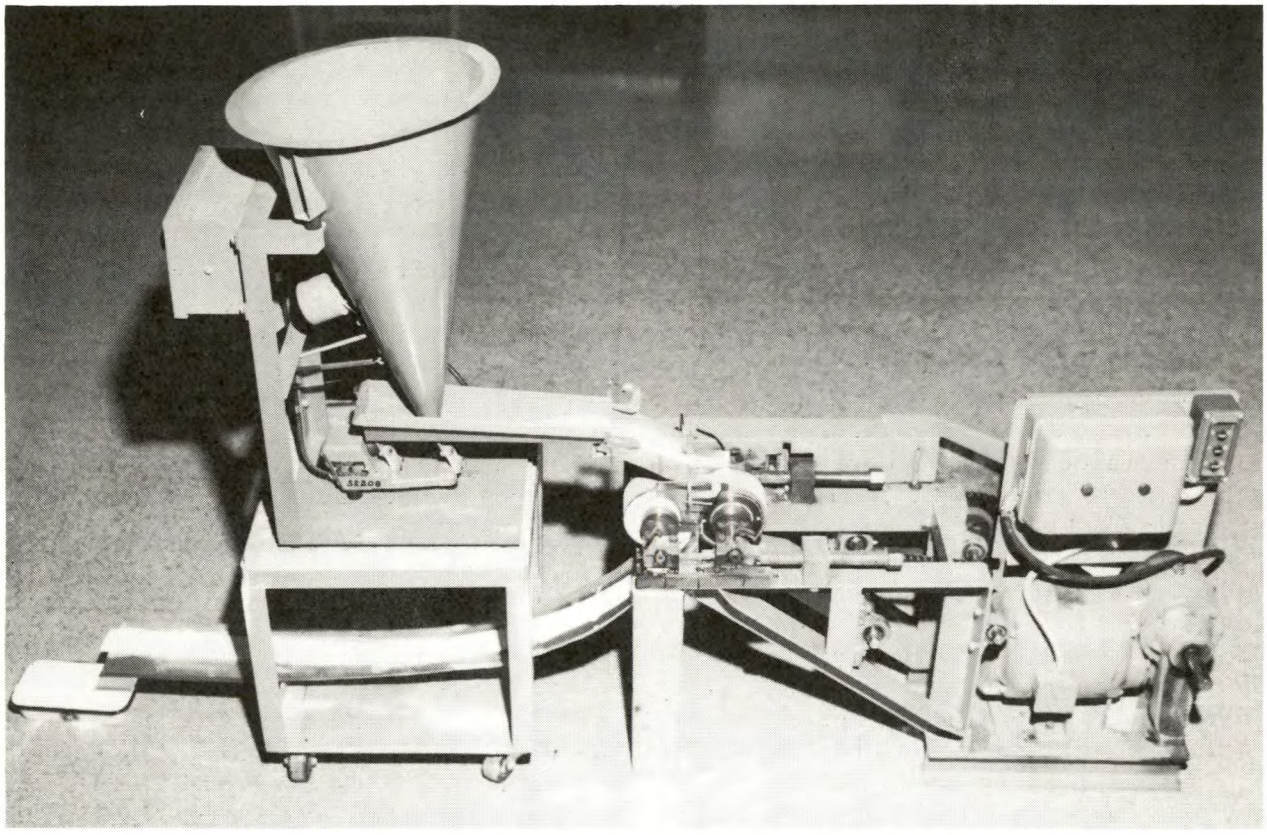


FIGURE 1. - Apparatus for Roll Forming Ceramic Powders Into Strip. Overall, the machine measures 1¼ feet wide, 2 feet high, and 4 feet long. Roll speed and roll size can be varied.

The powders were fed to the rolls through an open rectangular feed chute that contained no baffles or other regulating devices. The chute confined the powder to the roll entrance area and thus allowed control of feed rate into the rolls by regulation of the height of powder in the chute and the rate of feed to the chute.

Materials Processing

The stages of processing studied were powder preparation, mixing of batches, roll forming, and sintering. The compositions were tested in amounts of 300 to 500 grams, which produced from 3 to 10 feet of strip, depending on strip thickness, powder density, and batch size. The quantity of continuous strip made without interruption was a rough indication of the quality and processing uniformity under the conditions used. Admittedly, such small-scale processing is not a completely true assessment of processing uniformity, and some tests were repeated many times or were performed with larger batches.

Powders

The powders used in the compositions are given in table 1. Powder is used as a general, as well as a specific, term in this report and includes agglomerated materials such as pellets and granules. However, whenever granules were used, it is noted. Type A alumina was prepared by wet-ball milling 90-mesh, fused alumina to minus 325 mesh in a rubber-lined mill with alumina grinding media. The fused alumina was Norton Company blue label grade. Type B alumina was used as received and consisted of partially sintered granules of agglomerated finer particles. Types designated B, C, and D were Reynolds Metal Company grades RC152, RC152GF, and RC152DBM, respectively. Calcia-stabilized zirconia, GGC grade made by Zirconium Corporation of America, was used for tests as received or after fractionation or grinding to various sizes. For a few tests the same grade of zirconia provided as a minus 325-mesh powder was used after granulation. Other powders were Kingman potash feldspar and Edgar Plastic kaolin.

TABLE 1. - Powders and granules used in roll forming tests

Material	Type	Particle size[1]
Alumina:		
Fused	A	All minus 44 micron.
Sintered granules[2]	B	90 pct minus 100 mesh.
Sintered	C	50 pct minus 2 micron.
Do	D	70 pct minus 2 micron.
Zirconia	Calcia-stabilized	All minus 48 mesh.
Feldspar ($K_2O \cdot Al_2O_3 \cdot 6SiO_2$)	Whiteware	All minus 44 micron.
Kaolin ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$).	do	All minus 15 micron.

[1]See table 2 for more detailed particle-size data.
[2]Type B alumina consists of sintered granules as received.

Particle-size analyses of some of the oxide powders are given in table 2. For type B alumina the analysis is for the granules as received; the sizes of the agglomerated particles in the granules were not determined. The apparent sizes of sintered granules of agglomerated type D alumina and alumina-feldspar (AF3) are also given in table 2. Granules are similar to pellets except that sizes are generally smaller and surfaces more irregular. Like pellets or spray-dried powders, the granules are agglomerates of powder particles and have the free-flowing character needed for forming in powder-processing equipment. Table 3 shows the oxide content of the alumina and zirconia powders used in the strip. All grades of alumina contained over 99 percent Al_2O_3.

In most of the oxide mixtures, no inorganic additives were used that decreased the purity of the oxide powder. Slight increases in the impurity content of the oxides might result from the organic binders added for formability and bonding. However, spectrographic analysis of the powders and the sintered strip showed no appreciable change as a result of processing. The zirconium oxide as received contained the amounts of calcia and magnesia given in table 3 to partially stabilize the oxide structure in the cubic modification.

TABLE 2. - Particle-size distribution of some powders and granules, by cumulative weight-percent of particles finer than equivalent spherical diameter[1]

Material	Equivalent spherical diameter of particles									
	[2]295μ	[2]210μ	[2]147μ	[2]74μ	[2]44μ	20μ	10μ	5μ	2μ	1μ
Alumina A.....	-	-	-	-	100	91.7	52.7	29.7	11.6	5.3
Alumina B[3].....	-	99.3	91.2	22.6	2.2	-	-	-	-	-
Alumina C.....	-	-	-	-	-	98.6	95.8	83.4	12.3	5.3
Alumina D.....	-	-	-	-	-	-	100	95.8	37.8	5.0
Alumina D granules[3]....	88.0	62.8	40.6	14.4	5.6	-	-	-	-	-
Zirconia[4].....	96.5	79.7	59.2	35.8	22.4	-	-	-	-	-
Do.....	-	-	-	-	-	99.0	93.7	59.4	9.7	4.2
Feldspar.....	-	-	-	-	99	74.6	40.5	20.4	9.2	3.5
AF3 granules[3].....	95.2	82.8	64.4	44.2	21.9	-	-	-	-	-

[1]Equivalent spherical diameters determined by sedimentation or screen sizing.
[2]Corresponding micron and mesh screen sizes are 295μ (48 mesh), 210μ (65 mesh), 147μ (100 mesh), 74μ (200 mesh), and 44μ (325 mesh).
[3]Apparent size of agglomerated particles or sintered granules of Al_2O_3 or Al_2O_3-feldspar (AF3).
[4]Starting material used for fractionation or grinding.

TABLE 3. - Oxide content of powders, weight-percent

Material	Al_2O_3	SiO_2	Na_2O	CaO	MgO	Fe_2O_3	TiO_2	ZrO_2
Alumina A.....	99.2	0.30	<0.3	<0.1	<0.1	-	-	-
Alumina B, C, and D.....	[1]99.7	.08	.04	.02	-	0.03	-	-
Zirconia[2].....	.17	.40	-	2.90	.20	.01	0.11	96.0

[1]Calculated from impurity content. Includes traces of gallium and chromium shown in spectrographic analysis.
[2]Partially stabilized zirconia.

Mixing Procedure

Compositions for the roll forming tests were formulated by weight from one or more of the powders listed in table 1 and from a binder and water. Most of the compositions were prepared by what is termed the "normal" mixing procedure in this paper. In normal mixing the oxide powders and dry binder were combined and mixed in a blade-type mixer; then the water was added, and the composition was remixed. The binder and water contents were normally each less than 8 weight-percent of the mixtures. Mixing times were approximately 10 minutes for each stage. The consistency of the mixtures usually was similar to that used for dry pressing--slightly damp but flowable. Forming was done immediately after completing the wet mixing. This method was satisfactory for many compositions although not necessarily the best procedure.

In certain cases binders were added in a solution rather than as dry powders, especially when a binder was insoluble in cold water, when very fine powder was used, or when flexible strip with rubber binder was made.

Some of the more difficult to form compositions were mixed with excess water and were granulated or pelletized before being roll-formed. Most of the granules were made with a 40-mesh screen granulator and were then partially dried at room conditions before they were rolled into strip. In other tests the granules were sintered at various temperatures before use. The granulator had hard rubber-tipped blades that reciprocated in a trough and forced the powder-binder-water mixture through a screen in the bottom of the trough and onto trays for drying. Granules were placed in crucibles and sintered to develop a slight bond to permit satisfactory forming of the strip. Surface, or frictional, characteristics of granules are changed by the sintering, which also burns out the organic binders. Type D alumina granules were prepared from a mixture containing 5 percent gum arabic and 10 percent water and were sintered at 1,300° C for 3 hours. The AF3 granules were made from a mixture of 60 percent alumina and 40 percent feldspar bonded with 7.5 percent sodium silicate and were sintered at 800° C. Sintered granules were combined with binder and water, then mixed and roll-formed into strip. Combinations of sintered granules and fine-sized powders were used in some tests. Apparent particle sizes of the sintered agglomerates are shown in table 2.

Roll Forming

The forming process started when the freshly mixed powders were placed in the vibratory feeder, which was adjusted to provide a fairly constant head of powder approximately 1 inch above the rolls. The feed rate from chute to rolls was fairly constant, but it varied at the start and finish of each test. As a result the end portions of the strip, amounting to about one-fourth of the length, were usually discarded. When conditions were right, strip was formed by the combined frictional, gravitational, and compressive forces on the powder in the rolls, and then the strip was discharged along the curved offbearing chute at a rate approximately equal to the surface speed of the rolls. Normally, with the better compositions, continuous unbroken strip was formed, but other mixtures had poor green strength, and the strip broke into 1- to 6-inch sections. The strip was usually allowed to air dry for a few hours before cutting, trimming, or handling. Further drying at 110° C followed or preceded the cutting of sample shapes from the strip. The most common sample dimensions were 1-1/2 by 2-1/2 inches, but other sizes were used for some tests. Many compositions did not form strip, and others formed strip with visible defects. Defective strip was discarded except when information on density or sinterability was required.

Roll gaps were adjusted by removable gages, and initial tests on each composition were made using a roll opening of 0.04 inch. Readjustment of the roll gap was made when necessary following the initial test. The thickness of the strip was usually slightly more than the set gap because of slack in the roll mechanism and spring of the shafts. This spreading apart of the rolls was greater when roll gaps were less than approximately 1 percent of the roll diameter (0.05 inch), because pressures were higher when powders were compressed enough to make strip thinner than 0.05 inch. Nearly all forming was done at roll gaps less than 0.1 inch, or 2 to 2-1/2 percent of the roll diameter, because this was close to the upper limit of formability. Various roll surfaces and speeds were investigated, but most testing was done at 22 rpm using rolls with a 32-rms machined finish.

Special strips that had a corrugated, or sine wave, configuration were made to demonstrate the versatility of the process. The best roll configurations and the best composition for forming corrugated strip were not investigated.

Porcelain strip was made from mixtures of alumina-feldspar, alumina-clay, and alumina-feldspar-clay. Some of the porcelain strip was rerolled in air atmosphere at temperatures of 1,200° to 1,500° C in rolls that were at a temperature of 750° C, using the method, equipment, and procedures described in a previous report (5). Hot rolling was used to control thickness, finish, and density. However, acceptable porcelain strip also was obtained by normal cold rolling.

Sintering

Selected alumina strip was sintered at three different temperatures, 1,450°, 1,650°, and 1,800° C. Generally, sintering temperatures and rolling temperatures were measured with platinum versus platinum-13 percent rhodium thermocouples and were accurate to ±10° C of the stated value. Above 1,650° C, temperatures were measured with an optical pyrometer and were accurate to ±15° C of the stated values. Part of the strip sintered at 1,450° C was resintered at each of the other temperatures. Sintering periods at maximum temperature were 4 hours at both 1,450° and 1,650° C and 2 hours at 1,800° C. Zirconia strip was sintered at 1,450°, 1,800°, and 2,000° C for periods of 4, 2, and 1 hour, respectively. Sintering of the various types of strip is not necessarily optimum as no special sintering additives or atmospheres were used, but results are comparable for specific types.

Alumina-clay samples were sintered at the same temperature as the alumina strip. Other kinds of porcelain strip were sintered at temperatures between 1,000° and 1,400° C. Strip was usually sintered in an oxidizing atmosphere in electric or gas-heated furnaces except for a few, which were vacuum-sintered.

Physical Properties of Strip

The bulk density of the strip was measured to delineate relative quality and processing effectiveness. Data were obtained by calculation from measured dimensions and weights, and a few densities were determined by the water displacement method. Tests made with samples of several compositions indicate that densities may be 2 to 4 percent higher for comparable samples when determined by the water displacement method. Relative densities of the strip were calculated as a percentage of either the theoretical density for single oxides or of the measured pycnometric density of oxide mixtures. Shapes of most mixed oxides do not have a theoretical density. Some data were obtained on shrinkage during sintering and on the porosity of the sintered strip. The data collected serve to compare the effectiveness of roll forming the powders rather than to indicate the best quality of strip that could be made from similar powders by more refined processing.

Microstructures of various types of oxide strip were studied using ceramographic techniques. Thermal etching of polished alumina and zirconia strips in

vacuum was effective in revealing details of microstructure. Alumina samples were etched at 1,500° and 1,600° C for 5 minutes at a pressure of 10^{-5} to 10^{-6} torr; zirconia was etched at 1,800° C under similar vacuum conditions. Surface films showing interference colors were assumed to be metals vaporized from the heater and were removed by a 1-minute wash in 20 percent hydrofluoric acid.

DISCUSSION OF RESULTS

Alumina Strip

Many of the results reported are for tests made with type B alumina granules (table 1) because forming was better with coarser materials. This was not unexpected as previous research (3-4) clearly indicated the difficulties in forming fine-sized powders. However, the finer powders are of interest because more rapid densification at lower sintering temperatures is possible than with coarser powders, and considerable effort was made to find means of forming the finer sized alumina powders in later work.

Binder and Water Contents

Several binders were tested to determine the most suitable for roll forming type B alumina under the same forming conditions. The conditions included dry mixing with binder, followed by wet mixing with water, and forming at a roll speed of 22 rpm with 5-inch-diameter polished rolls and a roll gap of 0.04 inch. The water content was adjusted during forming trials to the range apparently right for the binder tested. The results are reported for these conditions, and no assumptions should be made of the relative merit of the binders for other conditions.

Strip formed with either calcium alginate or cornstarch as binder did not have acceptable green or dry strength. Mixtures containing cornstarch were too fluffy for ease in processing, and calcium alginate was not soluble or gelatinous in cold and hot water. Strip made with Stractan and Jelltrate did not air-harden as well as strip made with some other binders. Methyl cellulose, recommended by a previous investigator (3), required the use of special mixing procedures to obtain acceptable strip, including use of a dilute methyl cellulose gel made with hot water and drying the powders to exactly the right moisture content to allow roll forming. With a spray-drying or granulation procedure, this would probably be a suitable binder, but it is not suitable with the mixing method used.

Gum arabic was the best binder tested, considering all aspects of making strip, including forming, drying, and sintering. Type B alumina strip formed well with a gum arabic content of 5 to 12 percent, had good strength when green and dry, and sintered extremely well. A considerable number of detailed tests were made with selected compositions that contained 7 percent gum arabic and 7 percent water. For production uses the disadvantage of gum arabic is its relatively high cost, especially with the amount required. Mixtures containing lignin sulfonate binder roll-formed well at low binder and water contents (3 to 7 percent). Either lignin sulfonate or methyl cellulose is

cheaper than gum arabic, but strip densification by sintering was less than with gum arabic. Table 4 shows the bulk density of type B alumina strip made with various binders after sintering at three different temperatures. In strip bonded with gum arabic, fewer defects were apparent than when other binders were used. Binders best suited for alumina mixtures were also found acceptable for porcelain and zirconia compositions. None of the binders was effective in forming strip with powders much finer than minus 325 mesh.

TABLE 4. - Effect of binder type on bulk density of type B alumina strip dried or sintered at various temperatures[1]

Binder used	Dried at	Sintered at--		
	110° C	1,450° C	1,650° C	1,800° C
7 pct gum arabic.....	49.7	53.0	79.1	85.4
5 pct lignin sulfonate....	42.3	58.0	63.1	74.6
7 pct cornstarch.....	47.3	53.0	70.5	78.4
3 pct methyl cellulose....	40.5	44.9	64.8	70.4
7.2 pct rubber.....	42.5	43.2	59.8	71.6

[1]Bulk density of strip in percent of the theoretical density of alumina (3.98 g/cm^3).

Other binders unsuitable for roll forming alumina strip were carbowax, triethylene glycol, and stearic acid. Clay (kaolinite or ball clay) added to other oxide powders allowed roll forming of the mixtures with or without organic binder, as discussed more fully under "Porcelain Strip."

Generally, a water content of 5 to 10 percent in the type B alumina mixtures allowed forming of acceptable strip. Less than 5 percent water did not usually allow consolidation of the powders, and over 10 percent water normally caused the mixtures to stick to the rolls. Properties of sintered alumina strip did not change significantly with variation in water content from 5 to 10 percent.

Flexible Strip With Rubber Binder

Alumina (and porcelain) strips that were very flexible in the green and dry states were made with rubber binder. Two types of natural rubber solutions were tried. One contained 48 percent solids in xylene and the other 12 percent solids in aliphatic naphtha. The xylene solution provided a better bond in the strip than the other solution. With strip containing less than 10 percent rubber, air drying was required to develop flexibility. Strip with approximately 17 percent rubber was flexible as rolled and when rerolled, became leatherlike. Most of the strip containing xylene-derived rubber could be easily cut or stamped into shapes. This was also true of green or partially dry strip made with gum arabic. Wet mixing followed by drying was required to obtain feed powders suitable for forming the strip with a high rubber content. Water was added to some of the mixtures to be roll-formed after part of the xylene had been removed by air drying. By this procedure alumina strip that was flexible when dried at 110° C was obtained with a rubber content as low as 7.2 percent. The flexibility of the strip was a function of the rubber content. Strip made from the aliphatic naphtha rubber solution was not flexible when dry.

Strip containing the lowest rubber content sintered, as shown in table 4, to low bulk densities. Sintering defects occurred in strip of higher rubber content, probably because the heating schedule did not allow sufficient time for slow combustion of the rubber bond. Advantages of flexible strip are that it is easier to store, cut, and stamp than strip made with other bonds. No serious difficulties that could not be resolved were apparent. Ignatiev and coworkers (6) recently showed similar favorable results in roll forming strip from zirconium carbide powders containing 1 weight-percent of natural rubber bond.

Strip Thickness and Roll Gap

Tests were made to determine the range of strip thickness possible with the equipment and conditions used and the effect of roll gap on formability and properties of the processed strip. Type B alumina containing 7 percent each of gum arabic and water was roll-formed in 3- or 5-inch-diameter rolls operated at 22 rpm. Table 5 gives the results of two test series in which the roll gap was varied from 0.02 to 0.08 of an inch.

TABLE 5. - Dried or sintered bulk density of type B alumina strip related to roll gap, strip thickness, and roll diameter

Sample	Roll gap, inch	Dry strip thickness, inch	Bulk density, percent of theoretical[1]			
			Dried at 110° C	Sintered at-- 1,550° C	1,650° C	1,800° C
3-INCH-DIAMETER ROLLS						
AG12A	0.02	0.037	50.0	57.2	([2])	([2])
AG12B	.03	.047	48.9	56.7	([2])	([2])
AG12C	.04	.051	46.9	55.2	79.9	88.2
AG12D	.05	.058	45.4	53.0	-	[3]77.9
AG12E	.06	.069	43.4	51.0	67.6	75.4
AG12F	.08	.084	39.4	43.4	62.8	72.6
5-INCH-DIAMETER ROLLS						
AG12K	0.02	0.04	49.7	[4]53.0	79.1	85.4
AG12L	.04	.054	42.2	[4]43.7	66.3	74.3
AG12M	.06	.070	36.9	[4]37.7	55.2	65.5

[1]Bulk density of strip in percent of the theoretical density of alumina (3.98 g/cm^3) after drying or sintering as indicated. Sintering periods were 4 hours at 1,450° and 1,650° C and 2 hours at 1,550° and 1,800° C.

[2]Strip was too friable to sinter at these temperatures.

[3]Vacuum sintered for 4 hours at 1,750° C.

[4]Sintered at 1,450° C instead of at 1,550° C.

Strip was considerably thicker than the roll gap at the smaller openings because of flexure of the roll assembly under the pressure of forming the powders. With the compositions and conditions used, the practical thickness limits were 0.04 to 0.08 inch. Thinner strip was too friable to handle easily, and strip forming was not continuous at gaps of 0.08 inch or larger. Other investigators of ceramic (3, 6) and metal (2) powder rolling had similar results: A generally optimum forming thickness is 1 percent of the roll diameter.

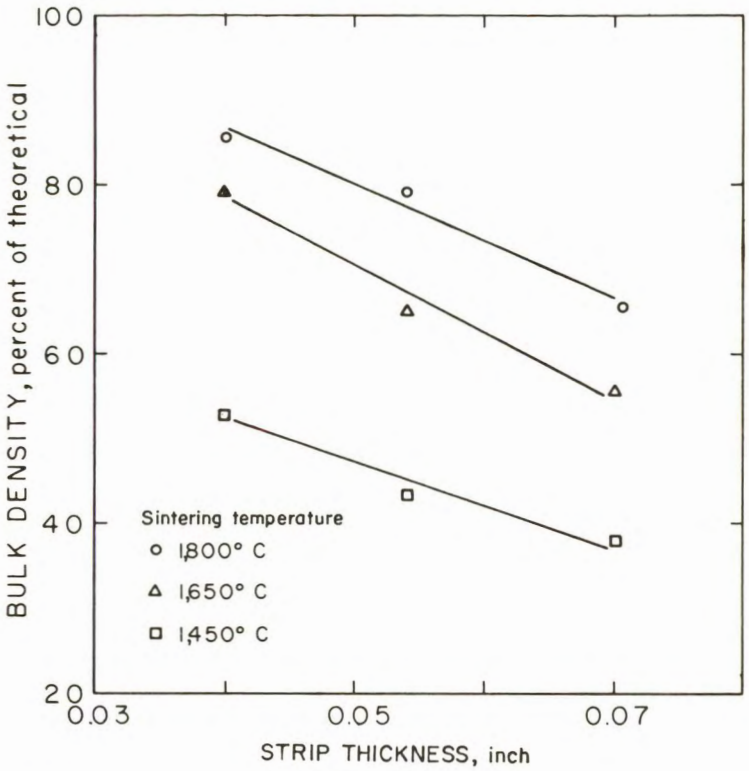


FIGURE 2. - Effect of Forming Thickness and Sintering Temperature on Bulk Density of Strip.

Bulk density increased as roll gap, and thus strip thickness, decreased, probably because of higher pressure and the smaller particle displacement required at smaller roll gaps. Density also increased with increased time or temperature of sintering. Differences in results (table 5) between comparable tests in the two test series reflect the change in roll diameter, which was 3 inches in the first series and 5 inches in the second. In later work, higher dry and sintered densities than shown in table 5 were obtained with alumina strip made from finer sized powders than type B alumina (tables 6 and 7), and thicker strip also was made. Figure 2 shows a plot of the data from the tests made with the 5-inch-diameter rolls.

Other investigators using large-diameter rolls have made thicker ceramic or metal strip, but as mentioned, 5 inches was the largest diameter used in the present work. The results showed that roll diameter and roll gap were important in governing the thickness of strip. The best strip made with the forming conditions tried was of good quality, as it was smooth, dense, and translucent after sintering at 1,650° or 1,800° C. The best conditions found for forming included a strip thickness of approximately 0.04 inch, a roll speed of 22 rpm, and a free-flowing alumina, such as type B, with additions of 7 percent water and 7 percent gum arabic binder.

The linear shrinkage of sample AG12L was typical of strip made from type B alumina: 3.0, 15.7, and 20.1 percent when sintered at 1,450°, 1,650°, and 1,800° C, respectively. Longitudinal and transverse shrinkage was similar, but shrinkage in thickness of strip was somewhat less, possibly because of the problems with the small dimensions involved or because powders were compressed more in thickness and thus had less shrinkage.

TABLE 6. - Effect of particle size and processing method on properties of alumina strip dried or sintered at various temperatures

Sample	Alumina type[1]	Composition variable or treatment	Forming quality	Bulk density, percent of theoretical[2]			
				Dried at 110° C	Sintered at--		
					1,450° C	1,650° C	1,800° C
AG2	A	Wet ground.....	Fair.....	66.3	70.6	78.9	81.2
AG26B	A	.....do.....	...do....	61.6	70.4	84.2	86.7
AG18A	C	12 pct gum arabic[3]	Poor.....	([4])	54.5	84.4	89.0
AG19	C	10 pct gum arabic[3]	...do....	([4])	69.6	95.2	96.2
AG18B	C	6 pct water[3].....	...do....	54.2	60.3	88.2	91.5
AG13B	C	8 pct water[3].....	...do....	57.8	64.6	95.0	95.6
AG13C	C	9 pct water[3].....	...do....	54.3	63.6	92.7	95.0
AG13D	C	10 pct water[3].....	...do....	54.0	61.6	88.4	([4])
AG13A	C	Granulated[3].....	...do....	54.5	([4])	88.5	89.9
AG13E	C	.....do.[3].....	Fair.....	51.0	63.3	92.7	95.2
AGN1	([5])	Dry ground[5].....	Good.....	70.9	67.8	73.2	74.9
AGN4	([5])	.....do.[5].....	...do....	70.1	69.3	73.7	78.1
AG12K	B	Coarse mix.....	Excellent	49.7	53.0	79.1	85.4
AG12L	B	.....do.....	...do....	42.2	43.7	66.3	74.3

[1]Specifications for alumina given in tables 1 and 2.
[2]Bulk density of strip in percent of the theoretical density of alumina (3.98 g/cm^3).
[3]Granulated, then partly dried before rolling.
[4]Not determined.
[5]Fused alumina, minus 90 mesh, dry ground with 1 pct naphthenic acid; AGN1 for 8 hours or to 62 pct minus 325 mesh, and AGN4 for 10 hours or to 85 pct minus 325 mesh.

TABLE 7. - Properties of dried or sintered alumina strip made from mixed powder sizes or presintered granules

Sample	Alumina type[1]	Treatment of powder	Forming quality	Bulk density, percent of theoretical[2]			
				Dried at 110° C	Sintered at--		
					1,450° C	1,650° C	1,800° C
AG16	70 pct B + 30 pct C	Mixed dry..	Good.....	49.3	54.3	74.1	89.0
AG14	50 pct B + 50 pct C	....do.....	...do....	45.5	50.5	72.6	86.5
AG17	30 pct B + 70 pct C	....do.....	Poor.....	-	-	-	-
AG2A	A11 D granules.....	Sintered granules.[3]	Good.....	51.0	57.5	73.4	83.4
AG2B	67 pct D granules + 33 pct D fines.	Granules[3]..	Fair.....	58.3	68.3	86.2	92.7
AG2C	80 pct D granules + 20 pct D fines.	....do.[3]...	Excellent	56.8	64.8	84.3	87.9
AG12A	B.....	As received	...do....	40.2	41.7	60.7	73.6
AG12K	B.....	....do.....	...do....	49.7	53.0	79.1	85.4

[1]See tables 1 and 2 for specifications on types of alumina.
[2]Bulk density of strip in percent of the theoretical density of alumina (3.98 g/cm^3).
[3]Screen-formed, 40-mesh granules sintered at 1,300° C before use in roll forming strip.

Roll Speed and Roll Surface

Strip of satisfactory quality was obtained at roll speeds between 10 and 60 rpm, or 13.1 to 78.5 surface feet/minute (SFM), with the 5-inch-diameter rolls. The

quality of strips as formed and after sintering was judged by visual examination and was considered satisfactory if samples were sound and free from visible defects. No obvious difference was noted in formability or quality of strip with this range of roll speed. Strip was not formed continuously at a roll speed of 100 rpm (131 SFM) because the gravitational flow of powder into the roll gap was too slow. It is interesting to note that these forming speeds are up to 13 times greater than those used by Fargo (3) and up to 23 times the speed used by Ignatiev and coworkers (6).

Limited tests of various roll surfaces were made. Strip formed well on rolls that were dry, damp, rosin coated, oiled, or heated. Grease and wax coatings prevented forming of strip from mixtures with the normal 7 percent water content. Talc coatings prevented sticking of mixtures too wet for normal strip processing. No specific tests were made to determine the best machined surface finish to provide optimum forming.

Changes in roll speed, roll gap, and roll surface were not effective in improving formability using powders that could not be formed under normal processing conditions. Lack of formability usually occurred with minus 325-mesh or finer powders, and no combination of roll-operating parameters was successful.

Particle Size

Numerous tests were made of alumina powders with the different particle sizes given in table 2. Formability was good with type B and marginal with type A, and the other powders were not formable except by use of sintered granules, which is discussed in the section on "Mixed-Powder and Sintered-Granule Compositions."

The most common defect in strip formed from the finer powders (types A, C, and D) in this investigation was V-shaped cracks extending across the strip. These finer sized materials occlude more air, which is not sufficiently eliminated before roll compression. If the air occluded and compressed on forming strip exceeds the pore space available at normal pressure, then the pressure differential may result in V-cracks or laminations. The rate of airflow out of the mixtures on roll compression must be proportional to the rate of strip formation and to the compression ratio. The compression ratio usually is higher with the finer powders, which have a lower powder density, but a higher roll strip density than coarser powders. Vinogradov and Fedorchenko (18) formed good strip using a partially evacuated chamber for feeding fine-sized metal powders, and this supports the assumption that gas pressure is one of the main difficulties. Another possibility is the larger surface area of finer powders, which can lead to higher frictional force than with coarser powders under similar rolling conditions. The higher frictional drag from the larger surface of fine powders may lessen the uniformity of flow and result in nonuniform pressure across the width of the strip. With small rolls, roll deflection may occur, which also would result in nonuniform pressure and possible variation in the exit speed across the strip. The V-shaped cracks probably result from combined forces caused by the foregoing reactions of fine powders and the rolls. Confinement of the strip edges during forming, mentioned by Fargo (3) and Ragan (15) for ceramic powders and by Blore and coworkers (2) for metal powders, might lessen the problem.

In addition, the lower flow rates of many finer powders cause intermittent feeding into the rolls. Free flow of powders is essential to continuous forming of strip, even at a moderate roll speed of 22 rpm, as enough powder must flow each minute to yield 29 linear feet of strip. The volume of powder needed is much larger than the volume of strip made because of the large compression ratios of fine powders. Some powders flow too freely, like a liquid, and will not compress into strip.

Various methods of preparing the finer powders (types A, C, and D) were used in an effort to make well-formed strip. Poor formability was evident by all methods used for forming these powders in the detailed preliminary tests. Normally the strip had a good finished surface, but it was not continuous because of numerous cracks. Nevertheless, some samples were cut from the best portions of most types and sintered to determine comparative physical properties. Table 6 gives the results with various fine powders and processing methods, along with the results for strip made from the coarser powders, which are shown on the last two lines of the table.

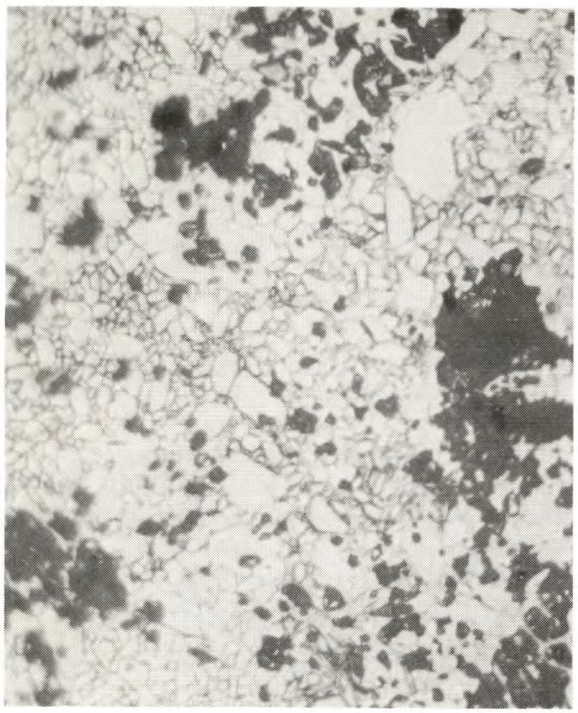
Most of the samples tested in table 6 formed generally unacceptable strip, but the data show the improved sinterability and high bulk density resulting from fine powders. Samples AG2 and AG26B were made from minus 325-mesh, wet-ground, fused alumina, which had marginal formability. Examples of the effect of granulating or pelletizing and partially air drying before forming are shown for mixtures AG18A, AG19, AG13A, and AG13E. Slightly improved forming was attained by some granulation procedures that did not include sintering of granules, but no method was completely satisfactory. Numerous tests were made with various binder and water contents without success. Dry-ground, fused alumina was used in mixtures AGN1 and AGN4, which allowed acceptable forming, but sintered densities were comparatively low even though the strip had a very high green density.

Corrugated Strip

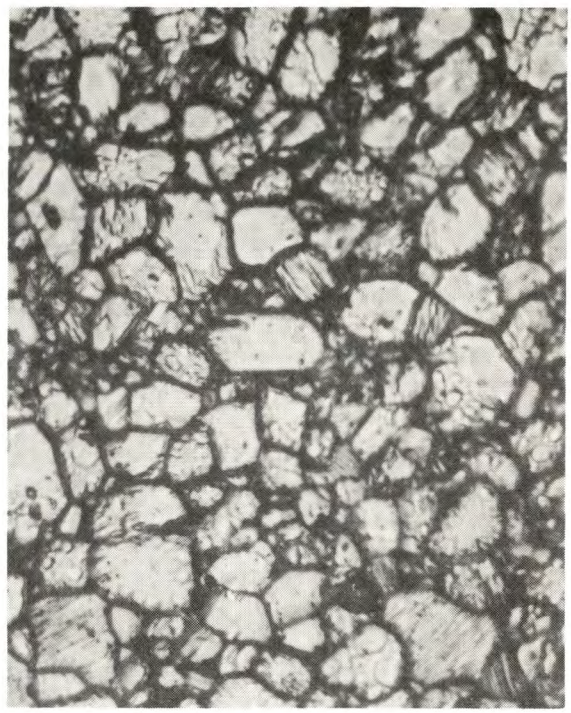
Corrugated strip was roll-formed from the AG12V alumina composition in shaped rolls to investigate the possibility of forming other than flat shapes by rolling ceramic powders. The process also suggests the possibility of making embossed or engraved patterns on ceramic strip by rolling. Only a few trials were made in forming the corrugated strip, and optimum forming and quality probably were not attained. However, no serious forming problems were encountered, and usable sintered strip was made. Combinations of flat and corrugated strip were assembled and joined into shapes by sintering. A sample of the corrugated strip is shown in figure 4.

Mixed-Powder and Sintered-Granule Compositions

Other tests were made using mixtures of different powders or of partially sintered granules and the same powder. Table 7 shows the results along with data for AG12A and AG12K made with powder B. The open porosities of the alumina strip sintered at 1,800° C were 15.5, 10.4, and 2.7 for compositions AG2A, AG2C, and AG2B, respectively. Porosity decreased with increased density or increased fine powders in the granule-powder mixture.

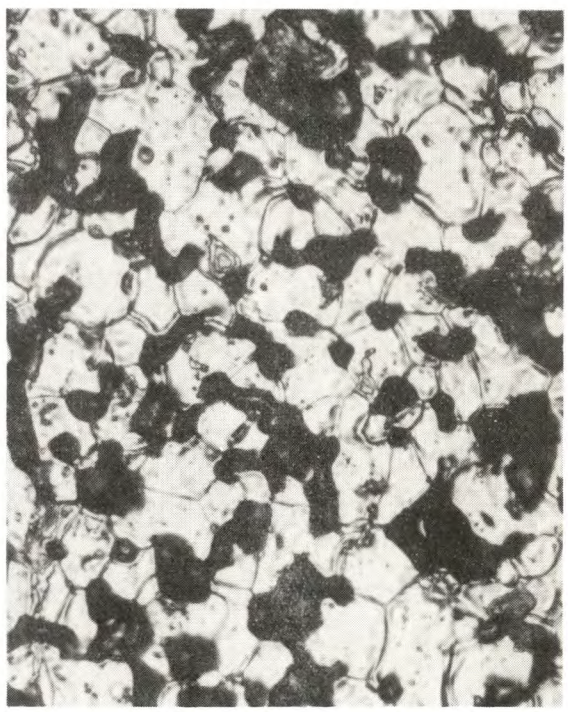


A – AG12K



B – AG19

C – AG16



D – ZG11

FIGURE 3. - Microstructures of Alumina and Zirconia Strips. (Magnified X 500.)

Sintered granules appear to be a promising means for making strip from otherwise nonformable fine-sized powders, but considerable work is needed to determine optimum granule size and content and the best sintering temperature. Considering both the final sintered quality and forming, composition AG16 made from mixed powders was the best alumina strip.

Microstructure of Strip

Figure 3 shows some representative microstructures of alumina and zirconia strips. Tables 6 and 7 give compositions and physical data on alumina strip shown in the illustrations. Thermal etching was used to reveal the structure of the polished samples. Figure 3A shows the structure of the normal-quality strip (AG12K) made from powder B after sintering at 1,800° C and thermal etching for 5 minutes at 1,600° C in air. The densest alumina strip made (AG19) is shown in figure 3B as sintered at 1,800° C and after vacuum etching at 1,500° C for 5 minutes. This sample had a density of 96 percent compared to 85 and 89 percent for strips shown in figures 3A and 3C, respectively. The AG19 strip was made from type A powder, but considerable grain growth occurred during sintering. Figure 3C shows the details of microstructure of AG16 strip made from a mixture of coarse and fine alumina powders after etching, as in figure 3A. Some of the larger dark areas in photomicrographs A and C are surface films instead of porosity, as acid washing was not used to clean these samples. Figure 3D shows the microstructure of a calcia-stabilized zirconia strip sintered at 2,000° C, then polished and vacuum-etched

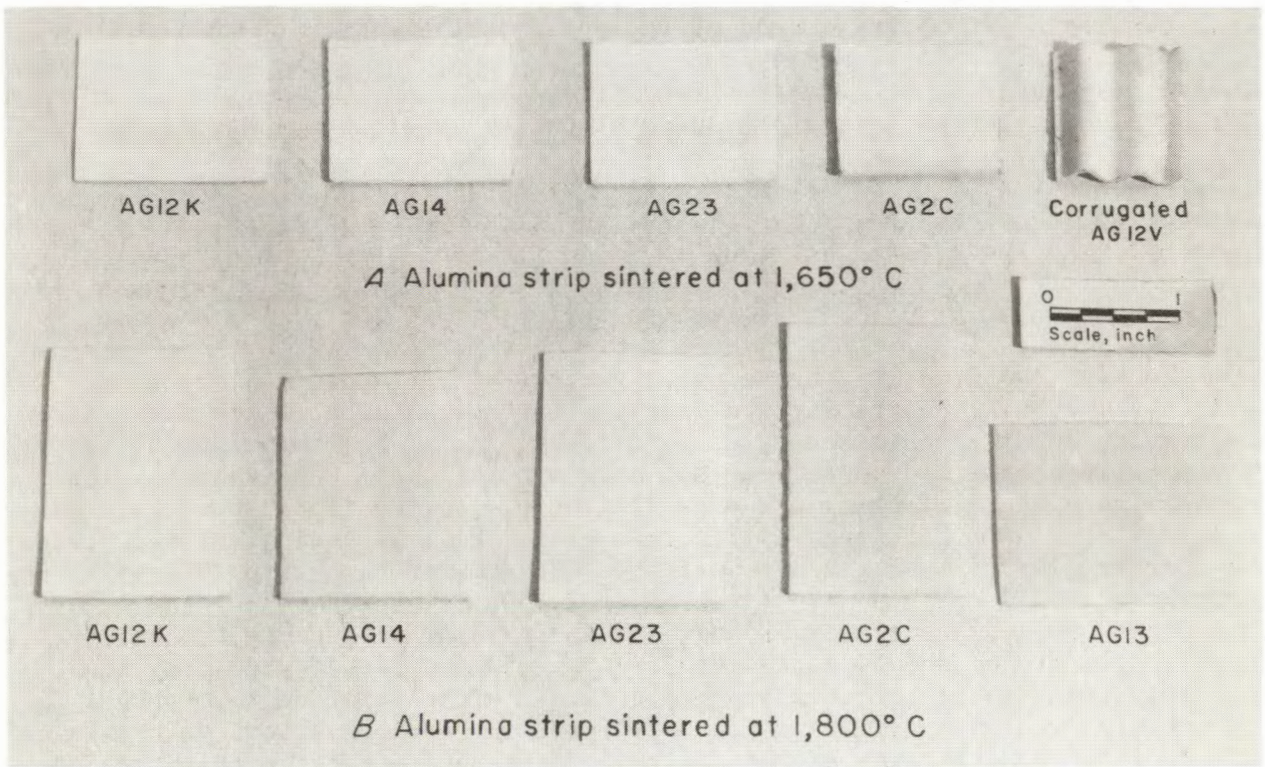


FIGURE 4. - Roll-Formed Alumina Strip.

at 1,800° C. (Results of roll forming this zirconia composition and others are reported in the following sections, and physical data are given in table 8.)

TABLE 8. - Properties of dried or sintered zirconia strip roll-formed from various powder sizes[1]

Sample	Particle size of powder[2]	Forming quality	Bulk density, percent[3]			
			Dried at 110° C	Sintered at--		
				1,450° C	1,800° C	2,000° C
ZG1	96.5 pct minus 48 mesh.	Good...	59.7	56.6	58.2	66.4
ZG2	96.5 pct minus 48 mesh.	..do...	64.2	63.0	64.4	66.8
ZG3	All minus 70 mesh.....	..do...	65.7	63.3	66.1	68.5
ZG4	All minus 100 mesh.....	..do...	63.7	59.7	64.1	65.5
ZG7	70 pct minus 325 mesh..	..do...	66.1	66.1	69.5	73.7
ZG8	86 pct minus 325 mesh..	..do...	70.6	68.4	74.6	76.5
ZG9	All minus 325 mesh.....	..do...	67.9	70.3	76.5	76.7
ZG10	86 pct minus 325 mesh..	..do...	69.2	71.4	74.1	78.7
ZG11	86 pct minus 325 mesh..	..do...	71.0	72.5	77.2	80.3
ZG12	70 pct minus 325 mesh..	..do...	68.2	69.5	73.2	77.7
ZG13	All minus 325 mesh[4]....	Fair...	([5])	60.9	([5])	86.0

[1] Compositions contained 3 to 4 pct gum arabic and 2 to 4 pct water, and strip was formed at a roll gap of 0 to 0.04 inch.

[2] Particle size fractions used (table 2) were either screened or milled from calcia-stabilized, minus 48-mesh zirconia powder.

[3] Bulk density of strip in percent of the pycnometric density of this calcia-stabilized zirconia of 5.48 g/cm^3.

[4] Laboratory-prepared, calcia-stabilized zirconia.

[5] Strip was friable and losses prevented sintering at all temperatures.

Samples of some of the alumina strip formed by rolling are shown in figure 4. The identities and properties of the strips were given in tables 6 and 7, except AG23, made from the minus 150-mesh fraction of type B alumina.

Zirconia Strip

The results of forming tests using various zirconia powders are given in table 8. The bulk densities given are relative percentages of the pycnometric density for calcia-stabilized zirconia of 5.48 g/cm^3. This mixed-oxide powder is not fully stabilized so it does not have a theoretical density. As with the alumina powders, forming was more difficult with the finest powders. Compositions such as ZG7 through ZG11 formed good quality, highly finished strip. The coarser mixtures had rougher surfaces, and some of the strip from mixtures ZG10 through ZG13 cracked when sintered at 2,000° C. Lower binder and water contents (2 to 4 percent) were required than for alumina because the higher specific gravity of the zirconia resulted in less surface area to coat per unit of weight. Good porous strip was obtained with sintering, but shapes did not densify even when formed by isostatic pressing for comparison tests, which indicates the powder lacks sinterability. Parameters for acceptable formability of zirconia strip were in the same range required for alumina strip. However, acceptable forming is difficult to judge as defects present after

sintering may or may not have resulted from forming problems. Narrower roll gaps of 0 to 0.02 inch, instead of 0.04 inch, were used to form ZG10, ZG11, and ZG12 strips, but neither the initial nor the sintered density markedly improved. Laboratory-prepared and finely ground stabilized zirconia used in ZG13 did not roll-form as well as the commercial grades, but it did sinter to higher density.

Table 9 gives the densities of mixtures of partially presintered granules and powders. Excellent forming was attained with the ZG21 and ZG22 mixtures, but densities were not high even when sintered at 2,100° C. The porosities of the strips were between 20 and 35 percent even when sintered at 2,100° C, but they did decrease in this range with an increase in amount of fines in the granule-powder mixtures. The quality of the strip was satisfactory, but further investigation is needed to obtain denser sintered strip. Samples of sintered zirconia strip are shown in figure 5. The round disk was stamped out of the partially dried strip.

TABLE 9. - Properties of dried or sintered zirconia strip roll-formed from presintered granules

Sample	Type mixture[1]	Forming quality	Bulk density, percent[2]			
			Dried at 110° C	Sintered at--		
				1,450° C	1,800° C	2,100° C[3]
ZG20A	All granules[4]...	Fair.....	53.6	57.4	67.3	67.9
ZG21	80 pct granules + 20 pct fines.	Excellent	56.8	59.8	69.5	77.0
ZG22	70 pct granules + 30 pct fines.	...do....	57.3	62.4	71.7	80.8
ZG23	60 pct granules + 40 pct fines.	Fair.....	60.8	64.8	74.5	83.2

[1]Mixtures contained 5 pct gum arabic and 5 pct water.
[2]Bulk density of strip in percent of the pycnometric density of this calcia-stabilized zirconia of 5.48 g/cm^3.
[3]Sintered in a vacuum of 10^{-5} torr at 2,100° C.
[4]Granules made from minus 325-mesh, calcia-stabilized zirconia with a 40-mesh screen granulator and then sintered at 1,300° C.

Porcelain Strip

Alumina-feldspar, alumina-clay, and alumina-feldspar-clay strips were roll-formed by the same procedures used for alumina strip. Data on some of these compositions are given in table 10. All the compositions shown were mixtures of presintered granules and unsintered fines. Composition AK1 contains type B alumina and clay. Granules made from type D alumina, then presintered at 1,300° C, were used in AK4, and alumina-40 percent feldspar granules presintered at 800° C were used in sample AF8. Clay, when used, was added separately in the noncalcined form. The data given are for 0.04-inch-thick strip, but thicknesses up to 0.08 inch were made. Granulation was necessary to obtain satisfactory strip from the fine-sized powders and to aid forming thicker strip. As shown, none of the strip sintered to high density at the temperatures used. The surface finish was not as smooth as most

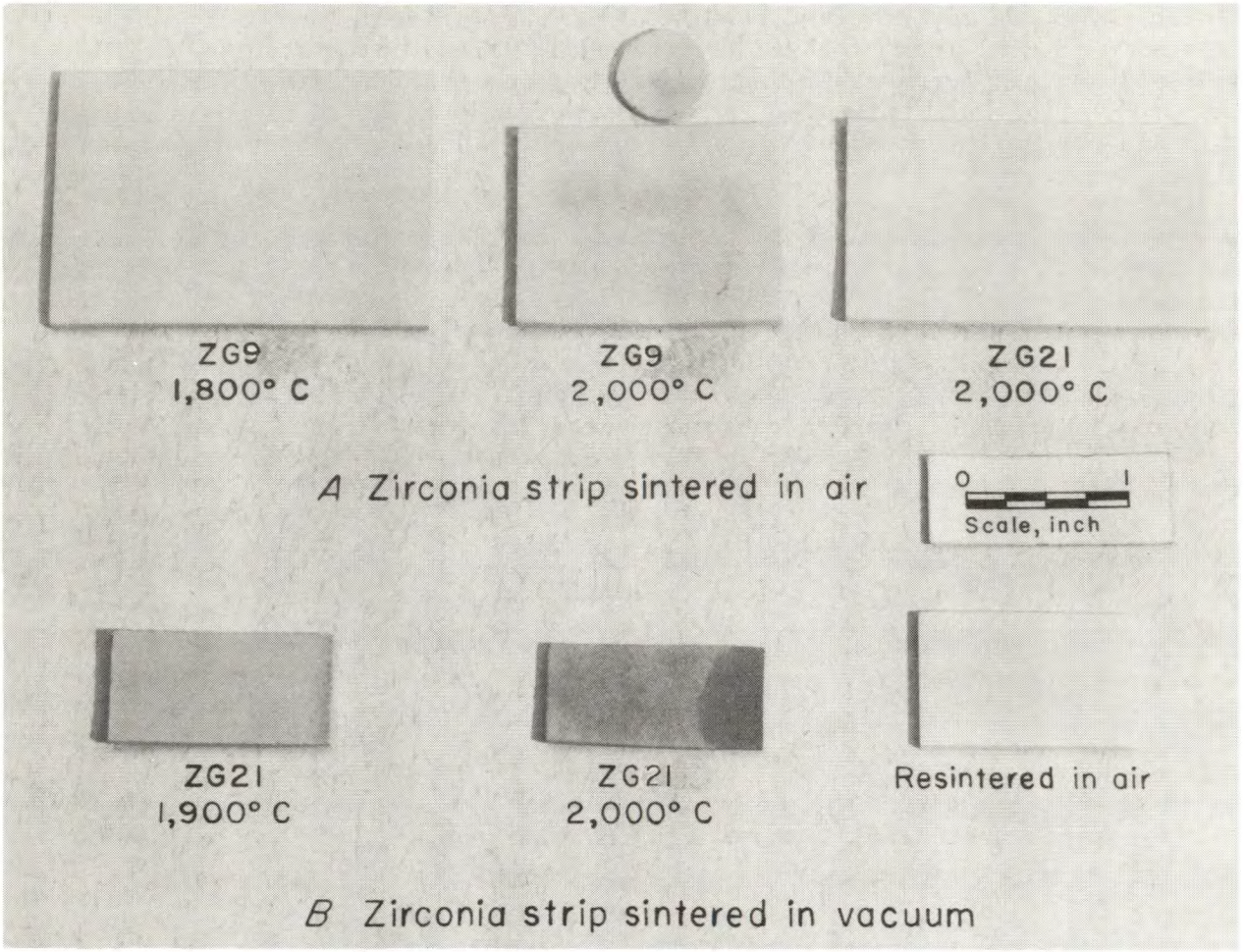


FIGURE 5. - Roll-Formed Zirconia Strip.

porcelains because of the inadequate sintering and the differential shrinkage between granules and fines.

TABLE 10. - Properties of roll-formed porcelain strip

Sample	Composition, weight-percent[1]	Forming quality	Sintering temperature, ° C	Bulk density			Surface finish
				Dry, g/cm^3	Sintered, g/cm^3	Percent[2]	
AF8	60 alumina-40 feldspar.....	Good...	1,250	1.70	2.74	88	Fair.
AK1	80 alumina-20 clay.....	Fair...	1,600	1.97	2.46	64	Do.
AK4	90 alumina-10 clay.....	Good...	1,600	2.02	2.61	68	Do.
AK4	.....do.....	..do...	1,800	2.02	3.44	89	Do.
AFC10	48.6 alumina-32.4 feldspar-19 clay.	..do...	1,250	1.56	3.32	77	Do.

[1]All strip formed from mixtures of presintered granules and unsintered fines.
[2]Bulk density of strip in percent of the pycnometric densities of samples.

Strip as thick as 0.15 inch was made by pressure-feeding powders to the rolls with a fitted tube and ram. Further study of pressure feeding would be worthwhile, especially if the powder was also under a moderate vacuum during the feeding process. The pressure-feeding method might bypass one of the main disadvantages of roll forming powders, namely the very large-diameter rolls required for making thick strip. (Estimated diameters are 25 inches for 1/4-inch-thick strip or 100 inches for 1-inch-thick strip.)

Hot Rolling Porcelain Strip

Strips of compositions AF8 and AFC10 made by cold rolling powders (table 10) were hot-roll reduced at 1,490° C to densify the strip and to improve its surface. This procedure parallels the successful method for rolling metal strip from powders. Table 11 gives the data on porcelain strip densified by the hot-rolling method described in a previous publication (5). Essentially, the method consists of passing the hot strip through rolls heated to about 750° C, or approximately one-half the strip temperature.

TABLE 11. - Properties of porcelain strip after hot rolling at 1,490° C in rolls at 750° C

Sample	Composition, weight-percent	Bulk density		Porosity, percent[1]	
		g/cm^3	Relative, percent[2]	Open	Total
AF8	60 alumina-40 feldspar.....	3.00	97	0.3 to 1.2	3.3 to 4.2
AFC10[3]	48.6 alumina-32.4 feldspar-19 clay.	2.91	96	.2 to 2.5	3.3 to 6.3

[1]Maximum and minimum values.
[2]Bulk density of strip in percent of the pycnometric densities of samples.
[3]Sintered density without hot rolling was 2.78 g/cm^3 at the same conditions, or 92 percent of pycnometric density.

Strip with a relatively smooth surface and high density was obtained by the hot-rolling method. The AFC10 strip had a density higher than the 92-percent relative density obtained by sintering the same strip at the same conditions without hot rolling. Thickness reductions were made in the range of 10 to 20 percent at rolling loads of 500 to 1,200 pounds. The porosity of the rolled strip varied considerably because of the differences in rolling conditions. No defects were noted in the rolled strip except for curvature from the rolls that was difficult to prevent when small and thin pieces were rolled. The advantages of hot rolling as a processing method would be precise control of strip thickness, as well as improved density.

SUMMARY AND CONCLUSIONS

Some of the critical parameters for the successful room temperature consolidation of discrete nonmetallic powders into strip were established by this research. The particle size of the powder, the type and amount of binder, water content, and roll gap strongly influence the quality and reproducibility of the roll-formed strip. Minus 100-mesh, 200-mesh, and 325-mesh powders were readily formable by normal processing methods. However, powders with

particles that were mainly less than 20 microns in diameter were not formable except when granulated and sintered to increase their effective size or when mixed with equal weights of granules or coarser powders. Satisfactory binders for roll forming included gum arabic, lignin sulfonate, and rubber dissolved in an organic solvent. With these binders the water content of the powders should range from 3 to 8 percent, depending upon the materials being roll-formed. The use of 8 to 10 percent clay obviated the necessity for any organic binder.

Roll gaps from 1/2 to 2-1/2 percent of the roll diameter produced strip ranging in thickness from 0.04 to 0.08 inch. The roll gap also influenced the bulk density of both green and sintered strip, as lowering the roll gap produced denser strip. Additional densification of the porcelain strip was accomplished by rerolling in heated rolls to attain as high as 98 percent of theoretical density. This method also was effective in altering the shape of the cross section of the strip. For example, flat strip could be formed into a corrugated shape by rerolling in heated rolls. Forming rates can be varied from 800 to 4,700 feet per hour.

Developments that make the roll forming process attractive for ceramic products are enumerated as follows:

1. The process is adaptable to continuous, automated forming, firing, and finishing.

2. The forming rate is high.

3. Powders of different particle sizes may be formed.

4. The process is capable of making strip in a wide range of thickness and densities.

5. The process has a potential of making flat and shaped strip.

Although much research remains to be done to optimize roll forming parameters and to delineate material properties for each application, this work has demonstrated the feasibility of roll forming ceramic strip.

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