Leco®
Met-Tips Ideas for Metallographic Procedures
No. 19

Metallographic Preparation of Ceramic and Cermet Materials

Ceramic materials are compounds of metallic or nonmetallic elements. There are many ceramics because there are many combinations of metallic and nonmetallic atoms. In general, ceramics are oxide-based materials, although the word "ceramic" has been expanded to include metal carbides and metal borides, as well as metalloids. Metalloids are elemental solids having only partial metallic characteristics; boron, silicon, and germanium being the more common ones. Single oxide ceramics are the most important of the currently used ceramics because of their greater resistance to oxidation at elevated temperatures. Single oxide ceramics are widely used in refractories for industrial furnaces and as fuels for nuclear reactors; certain mixed oxides ($BaTiO_3$, e.g.) are referred to as electronic ceramics and are used in fabricating capacitors, thermistors, and resistors.

Cermets are composites of ceramic material with a metal that are used for specific applications. The ceramic material can be a cladding on the surface of a metal or dispersed in a metal matrix. Most metal elements are capable of forming an oxide and thus would be considered a ceramic; however, the more common ceramics likely to be encountered in a metallographic laboratory are aluminum oxide (Al_2O_3), magnesium oxide (MgO), zirconium oxide (ZrO_2), beryllium oxide (BeO), uranium oxide (UO_2), or combinations of any of these. (NOTE: The metal beryllium and its oxides are health hazards!! All grinding must be done in the presence of water, and a mask should be worn over nose and mouth.) The scale that is formed on carbon steel alloys (FeO, Fe_2O_3, and/or Fe_3O_4), are ceramic materials; however, they are seldom referred to as such but simply as scale or oxidation product, unless they are used as semiconductors.

Following are the mineralogical hardnesses (MOH scale) and Knoop hardnesses (100 gram load) of the ceramics most likely to be prepared in a metallography laboratory.

Mineralogical and Knoop Hardness of Some Ceramics

Ceramic	MOH	Knoop
Al_2O_3	9	1600-3000 (a)
BeO	9	1100-1300
ZrO_2	8	1080-1520
UO_2	7	600
MgO	5	597-796

(a) depending upon density

Ceramics are inherently harder than metals but, at the same time, are more fragile, which makes them very susceptible to cracking and pullout (chipping) during the preparation stages. Because of these inherent characteristics, ceramic samples usually are kept as small as possible for metallographic preparation. Cracking is initiated during the sectioning stage, but can occur if improper mounting procedures are used. Pullout is a result of improper sectioning or a too severe grinding technique. Cracking cannot be rectified by subsequent grinding and/or polishing steps; pullout can, but it requires long, tedious intermediate polishing time.

The primary purpose of preparing ceramics for microscopic examination is to check for porosity that is present; however, the pullout that almost always accompanies a metallographic preparation makes the porosity studies somewhat dubious. As a consequence, porosity analyses are usually based upon density measurements (specific gravity), with microscopic examination used as a confirmation.

The manual preparation of ceramic material is a long and tedious affair and, more often than not, with only mediocre success. The long polishing times and heavy pressure required can be very frustrating, and as a result, a compromise is usually made between the amount of time being spent and the quality of finish.

Automatic grinding and polishing is highly recommended for those laboratories processing ceramic materials on a routine basis. Automation not only eliminates the frustration and tedium experienced with manual preparation, but can prepare multiple specimens in the same length of time required for one manually prepared specimen, and have superior results.

The techniques employed for the metallographic preparation of ceramic materials, while similar to those techniques employed for the preparation of metals or their alloys, are different enough that special precautions must be used to ensure samples are prepared to their optimum. The similarity of techniques lies in that the same basic steps of sectioning, mounting, grinding, and polishing are used, but each step requires a deviation from standard procedures.

The following techniques have been developed to maintain specimen flatness, keep pullout to a minimum, and certainly, to remove the tedium and frustrations usually encountered when metallographically preparing ceramic materials.

Sectioning

Whenever possible, sectioning should be done on a slow speed (300 to 500 rpm) cut-off machine using a diamond wafering cut-off wheel and oil as a lubricant. While cut-off machines with higher rpm (2800 to 3200) and larger diameter diamond cut-off wheels can be used, the slower speed cut-off machine will eliminate cracking of the ceramic material. The diamond blades used with slow speed cut-off machines are smaller in diameter (usually 4 or 5 inches) and much thinner (10 to 15 mils), thus keeping the material loss due to sectioning at a minimum. The size of the diamonds in the wafering blades should be 220 to 180-mesh size with 75 to 100% concentration for optimum sectioning; coarser mesh sizes have a tendency to fragment ceramic particles.

Abrasive wheel sectioning is not recommended, but if abrasive cut-off wheels are used, silicon carbide (SiC) wheels are better than aluminum oxide (Al_2O_3) wheels. The cutting edge of Al_2O_3 wheels will glaze very rapidly, and the cutting action is lost; SiC wheels will wear more rapidly, but a fresh cutting surface will be present at all times. Sectioning ceramics with abrasive wheels is slow, and depending on the size of the ceramic material, several wheels may be required to complete sectioning. The initial wheel-specimen contact should be very gentle to avoid wheel deflection and to establish a groove for the cut-off wheel to follow. Heavy pressure should not be used but rather a light, steady pressure, especially as the wheel approaches the exit point. Maintaining the same pressure as the abrasive wheel approaches the end of the cut will cause the ceramic to fracture the rest of the way, and a large burr will be present. Placing a steel sample with the ceramic sample such that the cut-off wheel comes in contact with the steel first will serve to dress the wheel and give better cutting qualities. Diamond cut-off wheels used with cut-off machines are more expensive than abrasive wheels on a one-to-one basis; however, diamond wheels do not reduce in size, can be used over and over again, cut faster, and produce a finer surface. The initial cost-per-wheel will soon pay for itself because of its longer life and superior cutting capabilities. If ceramic material is processed on a continuing basis, an investment in a slow speed cut-off machine (such as the LECO VC-50) is very essential.

The sectioning techniques for cermets are essentially the same as for ceramics, particularly if the ceramic material is dispersed in a metal matrix. If the ceramic is a clad on a metal surface, the sample should be oriented such that sectioning commences in the ceramic. If sectioning began in the metal, the ceramic would spall or fracture as the cut-off wheel neared the exit point.

Mounting

Unless the ceramic sample is of sufficient size to withstand the pressure required for compression mounting, mounting should be in one of the castables—such as epoxy or polyester resins with an accelerator (catalyst) to promote curing at room temperature. Long, thin ceramic samples, less than ¼-inch in thickness, always should be mounted in a castable mounting media. If samples ¼-inch or larger are mounted in a compression mounting press (using bakelite, epoxide, or diallyl phthalate mounting media), all burrs must be removed from the cut surface. A burr is usually present on one of the sectioned surfaces, the size of burr being dependent upon the sectioning procedures. A burr, regardless of how slight, will not allow the sample to lie flat, and the pressure used for compression mounting will cause the sample to crack. Removal of the burr is not as critical when using castable mounting media, but should be done in any case to establish a flat starting surface.

Any size ceramic sample containing large amounts of porosity should always be mounted in a long-cure epoxy mounting media where a vacuum can be pulled to ensure pore impregnation. This will eliminate the pore walls rounding during the polishing steps and also prevents grinding and/or polishing debris becoming entrapped in the pores. Cermets can be mounted in compression mounting media with relative safety.

Whatever type of mounting media is used, it should have a comparable abrasion rate—not hardness—as the material being mounted. Of the compression mounting media, diallyl phthalate has a comparable abrasion rate. Castable mounting media will usually have a faster abrasion rate than the ceramic material; however, there are several techniques that can be used to equate the abrasion rates. One is to strategically place "dummy" pieces of ceramic around the periphery of the mount; the other is to use alpha alumina powder (5 to 10 micron) as a filler which is mixed in with the castable and poured around the sample. Both methods are effective and will ensure flat interfaces.

Grinding (Manual)

Diamond grinding discs are recommended. The discs can be a mono-layer of diamonds, either metal or resin bonded, with a pressure sensitive adhesive back (PSA) on a metal plate, or diamond spots on a thick Mylar back. The PSA metal discs are placed on a polishing wheel and, once affixed, should remain in place until exchanged for a new disc.

A typical diamond grinding sequence is 40 or 30-micron, followed by a 15-micron. Wheel speed should be in the vicinity of 300 rpm and medium-to-heavy pressure used with a copious flow of water. If the ceramic has a very rough starting surface (such as a fractured face), grinding can start with a 125-micron diamond spot pattern disc to establish flatness. Instead of moving the sample around the grinding disc, better results will be achieved if the sample is held in one position but moved from periphery to the center of the grinding disc. The diamond discs will remove material very rapidly, especially with the coarser mesh sizes, and even pressure should be exerted to avoid a wedge-shaped mount.

Grinding with SiC is not recommended but can be done; however, the life of a paper is exhausted within 10 to 20 seconds for the coarser grits and much sooner with the finer grits, and several papers must be used for each grit size used. The tendency is to grind longer, but then the mounting media will be ground below the ceramic sample. If SiC grinding is used, best results are achieved with vertical disc grinding, 1200-1400 rpm, with water as a coolant; sufficient hand pressure cannot be brought to bear against the mount if hand grinding techniques are used.

Cermets that have the ceramic as a cladding should be held on the grinding discs such that the grinding path is from the ceramic to the metal substrate. This will eliminate the softer substrate smearing over the ceramic-metal interface.

Polishing (Manual)

After the final grinding step, the sample should be ultrasonically cleaned; or held under running tap water, rinsed with alcohol, and dried in a stream of air to remove grinding debris.

Usually, two polishing steps are required, depending upon the desired surface finish. The first step is with a 9 or 6-micron diamond compound impregnated on a silk cloth using an oil extender as a lubricant. This step will require the longest time. Polishing should continue until all pullout is removed, as finer micron sizes do not remove much material. The cloth must be as taut as possible with no wrinkles; loose surfaces will cause excessive wear or tearing of the cloth. Polishing wheel speed should be approximately 300 rpm, and as much hand pressure as can be brought to bear against the mount should be used; too light a pressure will be ineffective. Both hands are used to apply pressure.

The second polishing step is with a 3 or 1-micron diamond compound impregnated on a silk cloth. The same techniques are applied as with the 6-micron diamond polish.

The hands and wrists will tire very quickly because of the heavy pressure that must be applied. A periodic microscopic examination not only serves to check the degree of polish but gives a respite to the hands and wrists.

If desired, a final polish of 0.05-micron gamma alumina on a slight nap cloth (such as Lecloth) can be used to put harder microconstituents slightly in relief, which makes them more observable under the microscope. Polishing wheel speed should be approximately 150 rpm with heavy pressure. This step should be relatively short, one or two minutes at the most.

To determine if polishing is removing pullout, observe a certain area and monitor it during the first polishing step. If the sample is porous, the amount of "pores" will decrease until only the legitimate pores remain. If the mount is dense, all "pores" will be removed. Only when all pullout has been removed should the next finer polishing step by used. If pullout is excessive and the 6-micron diamond polish does not seem to be removing the pullout very fast, a 9-micron diamond polish can be used.

Table 1
Manual Preparation of Ceramic Materials

Grinding

Diamond Spot Pattern Disc	Time (sec.)	Wheel Speed (rpm)	Pressure (hand)
74-micron	300[a]	300	heavy
40-micron	200	300	heavy
20-micron	200	300	heavy
Polishing			
9 or 6-micron diamond/silk/oil	300	250	heavy
3 or 1-micron diamond/silk/oil	100	250	heavy
Optional			
0.05-micron Al_2O_3, H_2O	60	150	heavy

(a) Time is approximate.

Grinding and Polishing (Semi-Automatic)

Manual preparation of one ceramic specimen will require approximately 20 minutes; however, using semi-automatic procedures, six to nine 1¼-inch diameter mounts can be prepared in the same length of time, depending upon the size of the specimen holder and the equipment used.

Advantages to semi-automatic procedures, over-and-above the number of specimens that can be prepared in a given time, are less consumables used, flatter edges, less pullout, and less tedium and fatigue. The latter is due to pressure being pneumatically applied instead of hand pressure. A guideline of semi-automatic procedures is listed below.

Table 2
Procedures for Grinding/Polishing Ceramic Materials using Automation

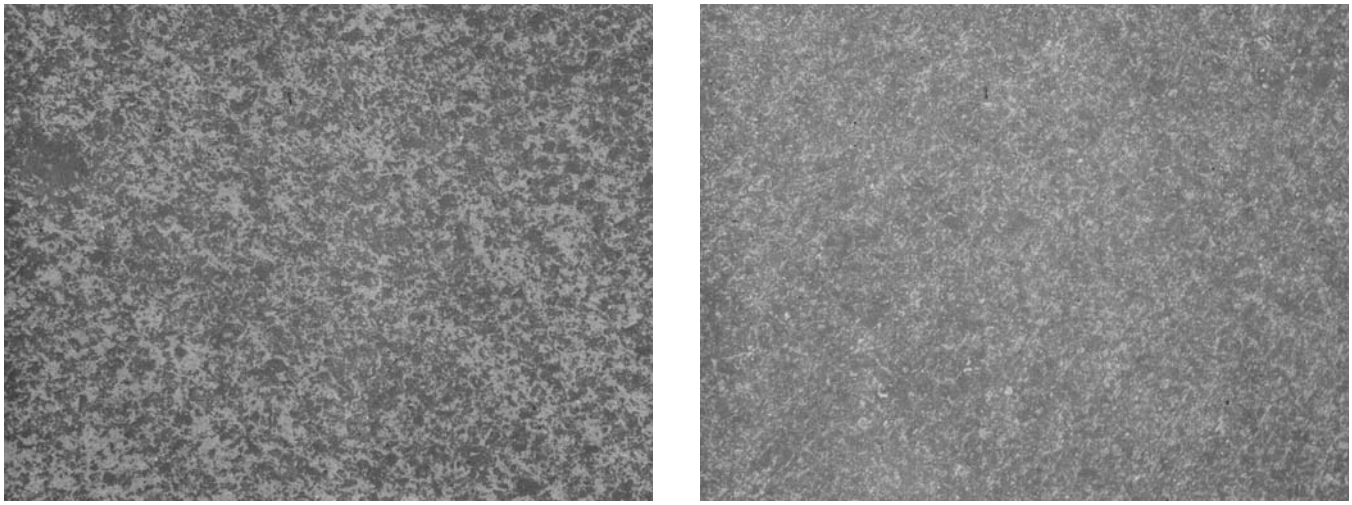
Grinding

Diamond Spot Pattern Disc	Time (sec.)	Wheel Speed (rpm)	Pressure (lb)
40-micron	120	200	50
20-micron	120	200	45
Polishing			
9 or 6 micron diamond/silk/oil	120	200	35
3 or 1 micron diamond/silk/oil	120	100	35

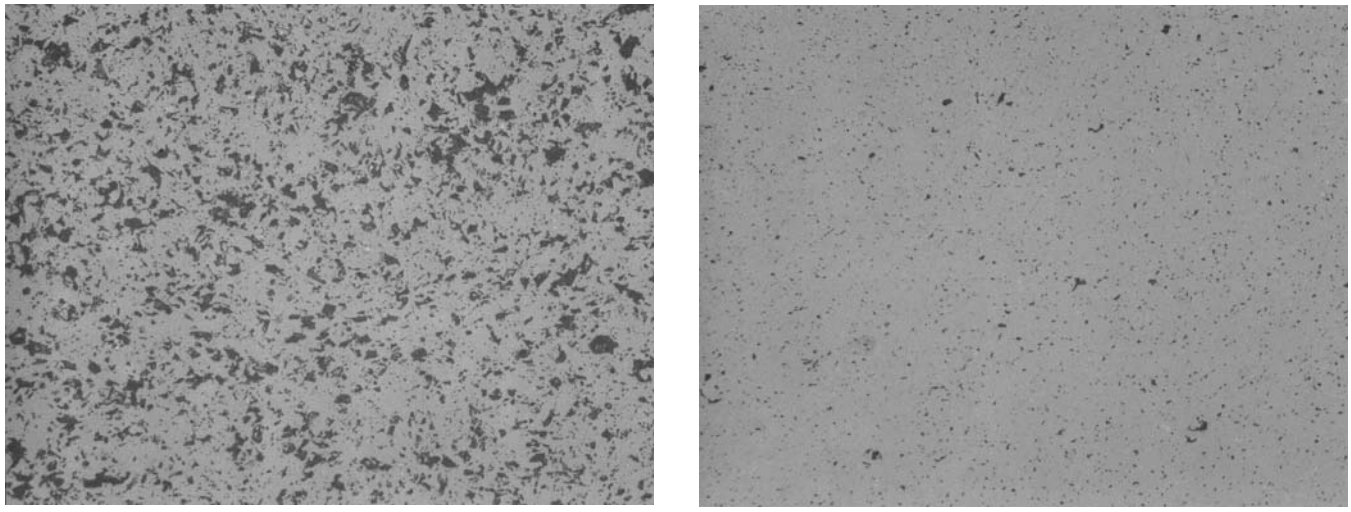
Although actual instrument time is eight minutes, it does not reflect handling time (i.e., changing wheels, ultrasonic cleaning, and a periodic microscopic examination to check the degree of polish). If handling time were to be included, total time would be approximately 20 minutes per specimen holder containing six or nine mounts.

Figure 1 shows a comparison between manual and automatic procedures used for metallographically preparing 99+% dense Al_2O_3. The same steps were used for each procedure; however, only one sample was prepared manually, while nine samples were prepared simultaneously using automation (such as the LECO GPX300 Automatic Polisher). The time spent on each procedure was approximately 20 minutes. Not only were nine samples prepared automatically in the same length of time it required for one manually prepared sample, but the degree of surface finish after each automated step was superior. The manually prepared sample could be prepared to have the same degree of surface finish as the automatically prepared one, but it would require a much longer time.

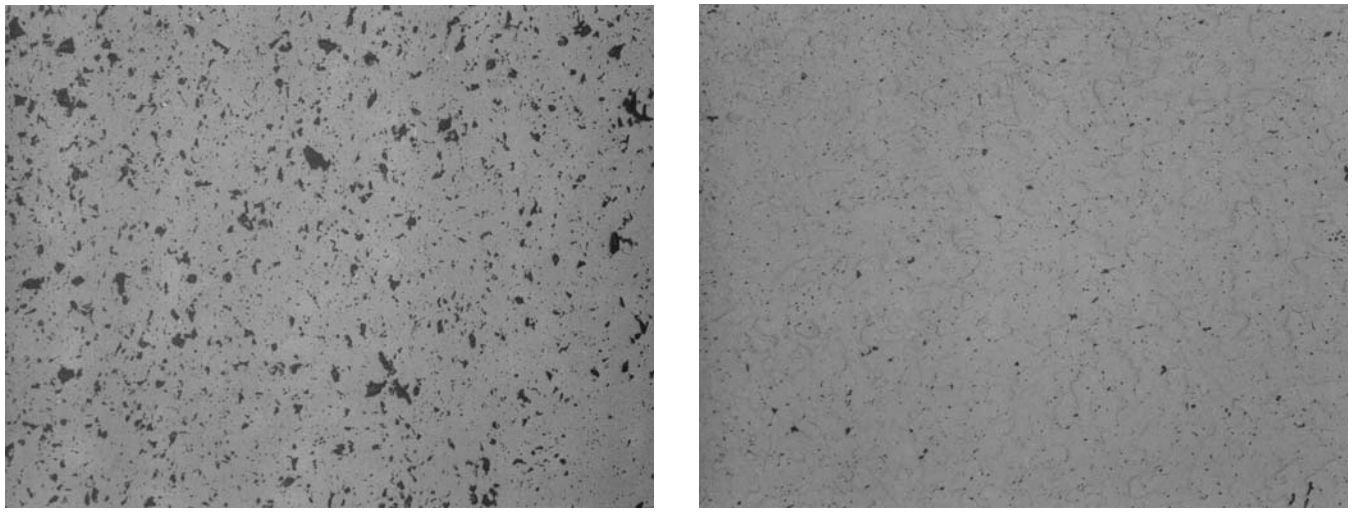
Manual **Automatic**



GRINDING—220-mesh diamond grinding disc followed by 15-micron diamond grinding disc.



INTERMEDIATE POLISH—6-micron diamond polishing compound on a silk cloth.



FINAL POLISH—1-micron diamond polishing compound on a silk cloth followed by silica polishing solution on a silk cloth.

Figure 1. Comparison between manual and automatic procedures; 99+% dense Al_2O_3; 100X magnification

Figure 2 compares the surface finish between manual and automatic procedures on 94% dense Al_2O_3 and 78% dense ZrO_2. The final polish was 1-micron diamond compound on a silk cloth, followed by a silica solution on a silk cloth.

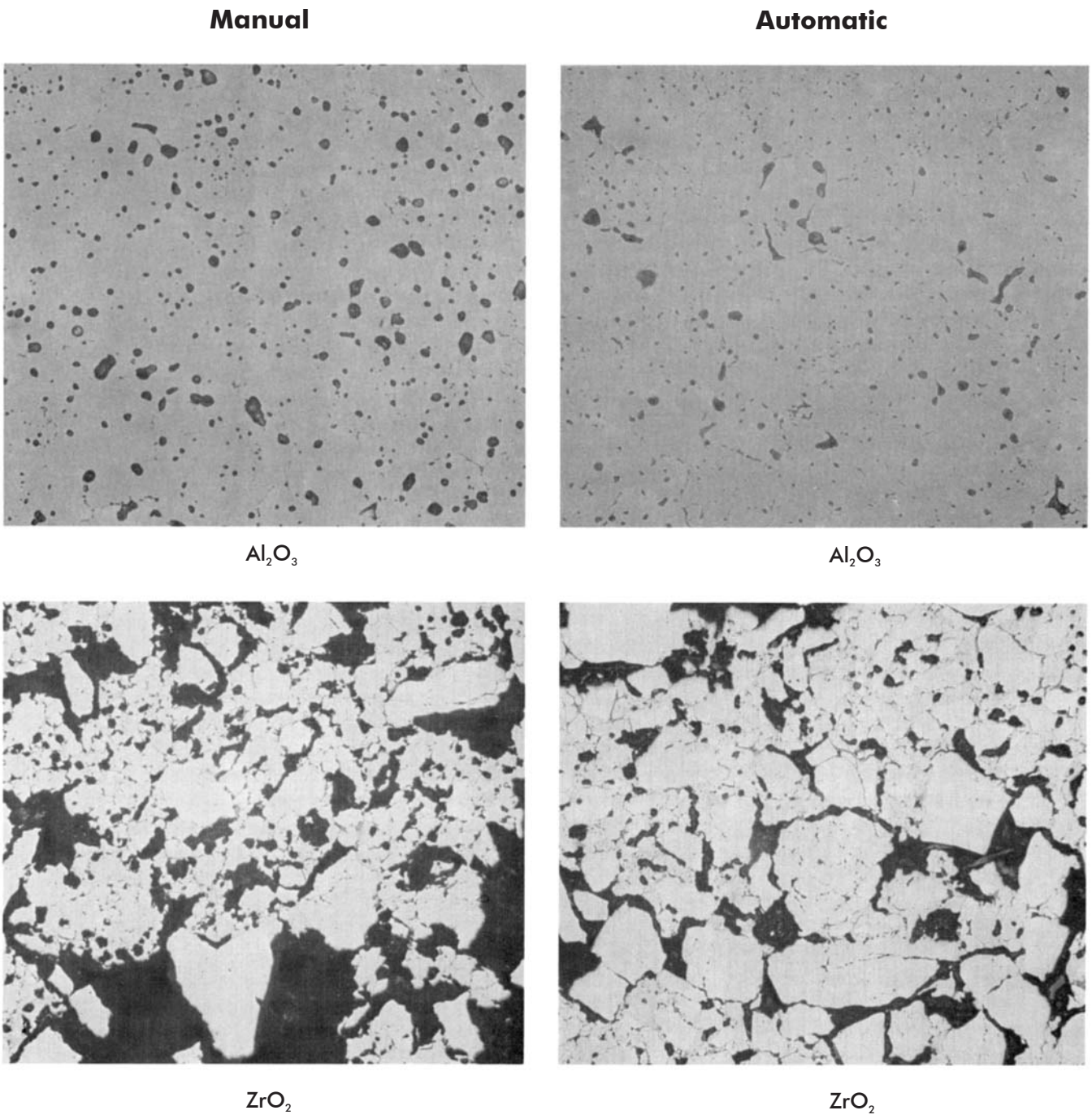
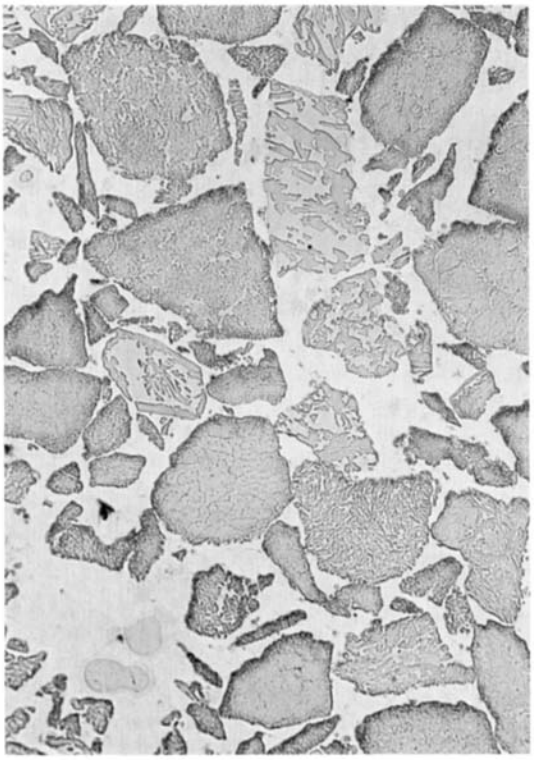
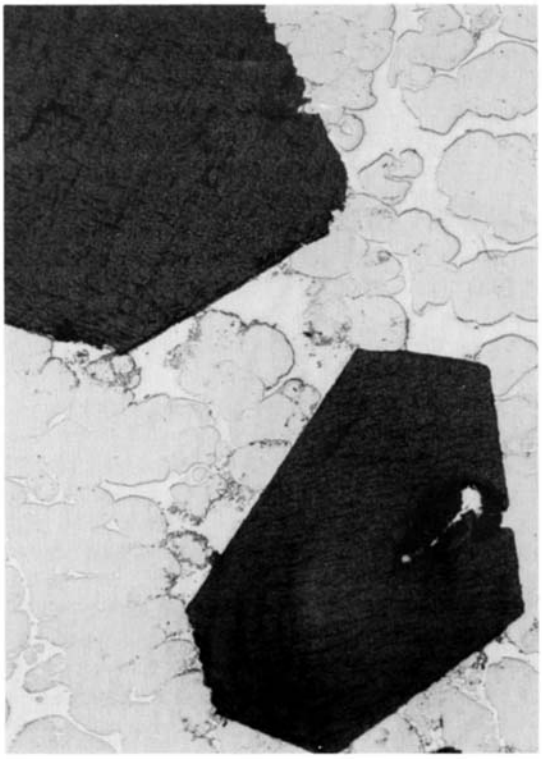


Figure 2. Comparison between manual and automatic procedures for 94% dense Al_2O_3 and 78% dense ZrO_2.

Figures 3 and 4 are examples of cermets which have been prepared automatically, and they represent some of the more difficult materials to prepare by manual procedures. Because of the great difference in hardness between the ceramic and matrix portions of the cermets, it is difficult to maintain the same plane of polish using manual means; however, with automation using pneumatic loading, the softer and harder constituents remain on the same focal plane.

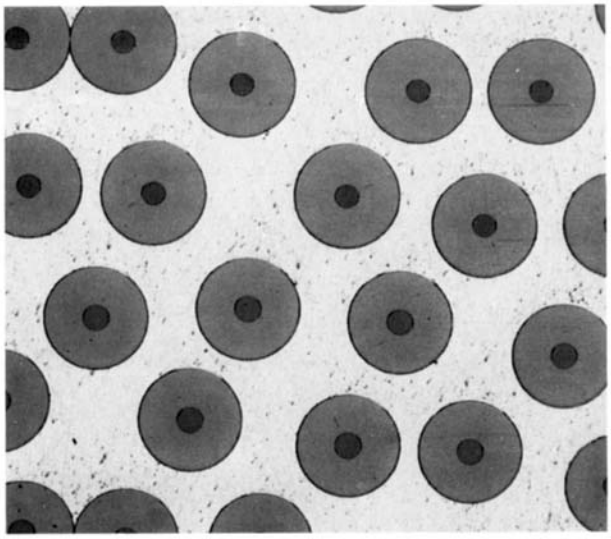


50X
WC in Aluminum Matrix

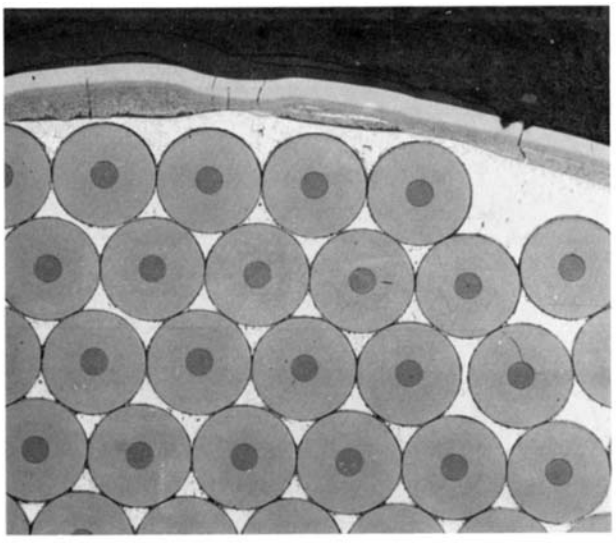


100X
Diamonds and WC in Aluminum Matrix

Figure 3. Cermets polished by automatic procedures.



100X
SiC Fibers in Aluminum Matrix



100X
B_4C Fibers in Aluminum Matrix

Figure 4. Cermets polished by automatic procedures.

The automatic procedures used for the preparation of the cermets illustrated in Figures 3 and 4 followed the outlines in Table 2, but were final polished with 0.05-micron gamma alumina on a medium nap cloth.

Etching

Ceramic samples are usually microscopically examined in the as-polished condition to check the inherent porosity; however, sometimes it is desirous to observe the grain structure. Following are etchants for the more common ceramic materials.

Aluminum Oxide (Al_2O_3)
Boiling phosphoric acid. Phosphoric acid contains water, and there will be a vigorous boiling action at first. Only after the vigorous action has subsided is the specimen placed into the etchant. Time will vary from one to five minutes. Rinse in water, flush with alcohol, and dry.

Magnesium Oxide (MgO)
90 cc H_2O
10 cc H_2SO_4
Use as a swab etchant by saturating a ball of cotton held in tongs. If etching is too light, specimen can be re-swabbed.

Beryllium Oxide (BeO)
20 g Ammonium Bifluoride
100 cc H_2O
Immersion. Bring etchant to almost the boiling stage. Time will vary between one to five minutes.

Zirconium Oxide (ZrO, ZrO_2)
50 cc distilled water
50 cc H_2SO_4
Immersion; one to five minutes; boiling.

Uranium Oxide (UO_2)
90 cc H_2O
10 cc H_2SO_4
Swab first, then immerse.

Barium Titanate ($BaTiO_3$, $BaTi_3O_7$)
10 cc HCl
3 cc HF
Immersion; 7 min. to 2 hours.

Uranium Nitride (UN)
30 cc Lactic acid
10 cc HNO_3
3 cc HF
Immersion; 1 to 5 minutes.

Uranium Carbide (UC)
30 cc Lactic acid
20 cc HNO_3
Swab; 1 to 3 minutes.

All etchants listed should be used under a fume hood, and adequate body and face protection should be exercised.

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LECO Corporation
3000 Lakeview Avenue • St. Joseph, MI 49085 • Phone: 800-292-6141 • Fax: 269-982-8977
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