LECO®
Met-Tips Ideas for Metallographic Procedures
No. 24

Automatic Specimen Preparation

The world's increasing advances in the metal, ceramics and microcircuitry technology and the need to know almost instantaneous answers have forced managers of quality control metallographic laboratories to take a long, hard look for a means to keep pace. Where it used to be five to ten specimens prepared daily, this now has increased to fifty or a hundred and sometimes even more. Quality control laboratories turning out 4,000 specimens per month no longer sounds astronomical; it is a fact. The manual, or hand polishing of specimens no longer can keep up with production needs and the manager must look to other methods such as machines for rapid specimen preparation. Automatic polishing: metallographers used to look in disdain at machines that attempted to do all or part of their work. And rightfully so. Up until the past several years, the automatic preparation of specimens usually required more time and effort than the final result was worth. Actually, there was no ideal automatic polisher. There were many types available, but many were considered a novelty and soon discarded in favor of manual preparation. Each had its own merits and shortcomings, and it was up to the metallographer to determine which type best suited his needs. Among a large number of types available were ones that used weighted samples held in an oscillating holder which in turn was motor driven across the polishing wheel. The specimens were free to rotate about their own axes due to the wheel rotation. Other types held the specimens rigid but allowed rotational movement of the specimen clamping device to follow the same pattern on the polishing wheel. Still other types used vibratory means where weighted specimens rotated around their own axes as well as around the periphery of the polishing area. All worked to a certain degree; edge flatness was usually maintained, the amount of disturbed metal was reduced and they freed the operator to do other things. However, they still took too much time, particularly in vibratory polishing. The idea was there—weight—but unfortunately not enough weight could be brought to bear without stacking weights on top of one another. This, however caused a shift in the center of gravity which in turn resulted in an uneven polished specimen. For these reasons, metallographers were reluctant to try semi-automatic methods for specimen preparation.

An automatic system for rapid preparation of multispecimens has been designed by the LECO Corporation in St. Joseph, Michigan. Without going into a lengthy technical description of the system (such information is available from the LECO Corporation) it is felt a brief description is pertinent. The automatic system incorporates the latest technology in that microcomputers, called microprocessors, are used to program selected parameters such as polishing wheel speed, time, pressure (in psi), type of liquid being dispersed as lubricants, frequency of dispersion and in some units, spindle speed, direction of spindle rotation, stock removal, either in inches or millimeters, and oscillation.

A large selection of specimen holders will accommodate unmounted samples having non-uniform morphologies as well as the universal sizes of mounted specimens. A specimen leveler insures all samples start at a common plane.

Samples obtained from an abrasive wheel sectioning operation can go directly to the polisher for fine grinding; rough surfaces such as flame cut or as-cast surfaces may require the grinder for rapid metal removal and to establish coplanarity between the samples and sample holder prior to going to the polisher. Since the majority of specimens processed through a metallographic laboratory have abrasive wheel cut surfaces, this article reports data generated from the polisher.

Automation in the laboratory is viewed in different aspects by different people. The manager: (1) cost; not so much the initial cost of the instrument but the cost of consumables used, (2) how much downtime can be expected, (3) how does the quality of the finish compare to the method presently used (manual preparation), (4) how quickly can specimens be prepared, (5) are the results reproducible? By the metallographer; (1) how will it affect my job (security), (2) is this going to take the art out of metallography, and (3) how long do I have to play with this before I can convince my manager that the old way is the best way? True, these latter questions are asked more frequently by the research metallographer than by the metallographer in a quality control laboratory where rapid specimen preparation is essential to meet production needs, but the skepticism is there, if not actual fear. No one likes the idea of being replaced by a machine. However, the fact is that one is not replaced by a machine; the machine is used as an extension to the individual.

The manager's questions can be answered in a few sentences. Over and above the cost of the instrument, the cost of consumables is usually drastically reduced. More specimens can be processed through a single grinding/polishing step, and many grinding steps can be successfully eliminated. Downtime is negligible other than routine maintenance. The quality of finish usually will surpass that obtained by manual means. More specimens can be prepared in a given time, and the reproducibility is excellent once the optimum parameters are established for that particular alloy system being prepared.

Answers to the metallographer's questions are more psychological in nature; one is dealing with emotions rather than in dollar and cents. The metallographer must be convinced that his job security is not threatened but quite the contrary, it is increased. True, metallography is both an art and a science, but the art need not be removed because a machine is doing the routine work. The standard of perfection the metallographer establishes for himself in manual preparation will be carried over to automatic procedures and his initiative expanded. As far as the metallographer wanting to revert to manual preparation, once he is convinced the machine is not going to replace him but will increase his efficiency and at the same time eliminate the tedium involved in manual preparation, he will soon begin to look for ways to use the machine to his advantage.

A competent metallographer can prepare a specimen manually form the mounting stage, ready for microscopic examination in the etched condition in approximately fifteen minutes, depending upon the alloy being prepared. An automatic polisher can do up to twelve specimens in the same time and have flatter edges, less disturbed metal and use less grinding/polishing steps than in manual preparation. For example, the accepted procedures used in manual preparation consist of a series of grinding steps commencing with a 180 SiC grind, followed by 240-, 320-, 400- and 600-grit grind; intermediate polishing with a 6-micron diamond compound followed by 1-micron diamond polish, then concluding with a final polish consisting of sub-micron polishing abrasives. This is a total of eight grinding and/or polishing steps.

An automatic polisher eliminates two or three grinding steps and one intermediate polishing step with more than satisfactory results. The procedures established for ferrous and nonferrous alloys processed through the polisher are basically the same; the deviation being mostly in the final polishing abrasive used.

The procedures outlined in the table below have been successfully used for low and medium carbon steels, gray and nodular cast iron, free machining steels, super alloys, heat resistant alloys, carburized steels, titanium alloys, copper alloys, aluminum alloys, microcircuit boards, capacitors and resistors, plated samples, samples with oxidation on the surfaces, in both the mounted and unmounted condition.

Actual instrument time shown in the above table is eight minutes for processing one specimen holder through the grinding steps, intermediate and final polishing steps. The choice of specimen holder used could have held six, nine, ten, or twelve mounted or unmounted specimens. The time does not reflect handling time, i.e., changing wheels, ultrasonic cleaning between the grinding and polishing steps, and perhaps a periodic examination with a microscope to check the degree of polish. If handling time were to be included, the total time would be approximately fifteen minutes.

In many instances, for example carbon steel alloys, the initial grinding step commenced with a 240-grit SiC grind followed by a 600 grit SiC grind, thus eliminating yet another grinding step. In another case, twelve mounted titanium alloy specimens went from the 600 grit SiC grind directly to 0.05-micron gamma alumina with excellent results. The 600 grit scratches were effectively removed and etching showed the specimens to be virtually free to disturbed metal.

Guidelines for grinding/polishing specimens using automatic preparation

Grinding

SiC GRIT SIZE	POLISHING WHEEL TIME (SEC)	SPINDLE SPEED/ SPEED (RPM)	DIRECTION (RPM,CW/CCW)	OSCILLATION (Y/N)	PRESSURE (PSI)
180	60	300	100/CW	Y	45
320	60	300	100/CW	Y	45
600	60	300	100/CW	Y	45

Polishing

1 Micron Diamond Compound/ Red Felt Cloth/ Microid Extender	240	250	75/CW	N	40
Colloidal Silica/ Imperial Cloth/ Water	60	150	75/CW	N	10

Any metallographer who has prepared low-carbon steel alloys consisting primarily of ferrite with a few colonies of pearlite recognizes the fact that the first time the specimen is etched after the final polish, the amount of disturbed metal is quite noticable. Figure 1 illustrates an SAE 1010 resulfurized steel that was prepared manually through all the grinding stages commencing with a 180 SiC grit grind followed by 240-, 320-, 400- and 600-grits; intermediate polish with 1-micron diamond compound followed by a final polish using 0.05-micron gamma alumina and etched after microscopic examination showed the final polish to have effectively removed all traces of a diamond polish. As can be seen, the amount of disturbed metal in the ferrite grains from the first etch is very extensive; also directionality can be seen that has persisted from a grinding step even through microscopic examination after the final polish showed no evidence of grinding scratches.

Nine specimens of the same alloy were processed simultaneously through the automatic polisher using the parameters outlined in the table. Figure 2 shows the first etch on one of the specimens and as can be seen, the amount of disturbed metal has been greatly reduced. The pearlite colonies are cleaner and the lamellae are resolvable at higher magnifications.

Not only did the automatically prepared samples exhibit virtually no disturbed metal, but nine specimens were prepared in approximately the same length of time that was required to prepare one sample manually. In regard to the manager's question of consumables costs, only one disc was used for the manual preparation but for only one specimen, and it is extremely unlikely that a metallographer would save or retrieve used discs for other grinding operations.

Figure 3 illustrates a free-machining steel prepared by automatic polishing. The surfaces of the nine samples were originally band-saw cut surfaces and were extremely rough. The automatic grinder was used for 60 seconds to obtain coplanarity between the specimen holder and the samples before going to the automatic polisher for fine grinding and polishing. The fine grinding steps consisted of a 240- and a 600-grit SiC grind before an intermediate polish of 1-micron diamond compound on a red felt cloth, followed by a final polish using 0.05 micron gamma alumina on a flocked twill cloth. Total time for preparing nine samples was four minutes instrument time. The photomicrograph showing the etched condition are from the first etch.

Nine, $1^1/_4$-inch mounted specimens of a plasma coated, nickel base superalloy were prepared simultaneously; the grinding steps consisted of a 180 SiC grind for one minute and 30 seconds each for a 320- and 600-grit grind. The final polish was with 0.05-micron gamma alumina for 60 seconds. The edges of the specimens were extremely flat and all traces of the 600-grit scratches were effectively removed.

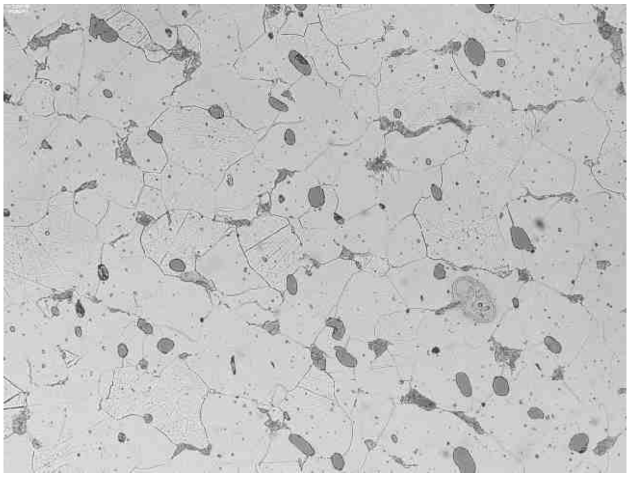

Figure 1: SAE Low-Carbon Resulfurized Steel Manually Prepared and Etched with 4% Picral, 500x

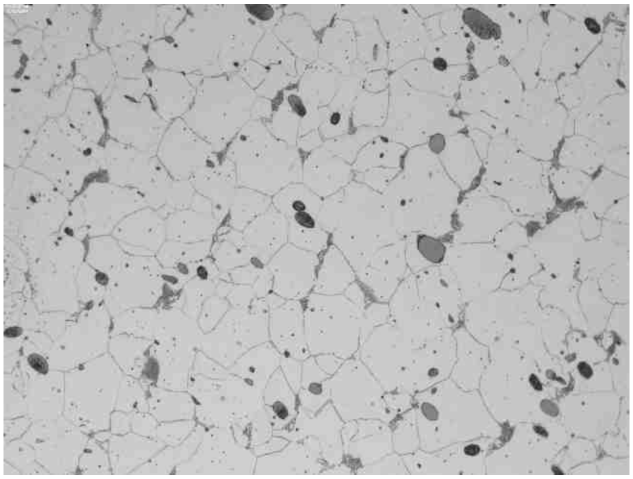

Figure 2: First Ected on an SAE Low-Carbon Resulfurized Steel Automatically Prepared and Etchant with 4% Picral, 500x

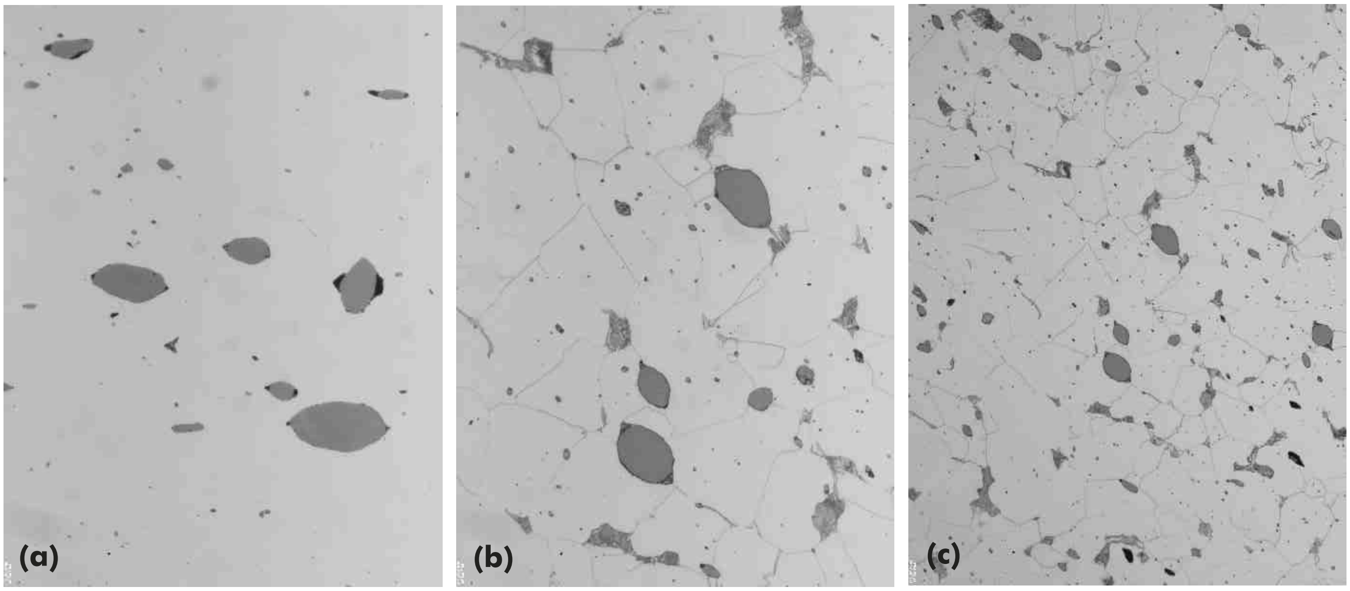

Figure 3: Free Machining Steel, Automatically Prepared.
(a) 1000x, As-Polished, (b) 1000x, Etchant: 2% Nital, (c) 500x, Etchant: 2% Nital

Gray cast iron and nodular cast iron lend themselves extremely well to automatic preparation with no sacrifice in quality; in fact kish graphite, which is usually difficult to maintain in its entirety during manual preparation without involving long final polishing times, came out flat and intact. Figure 4 shows gray cast iron prepared as follows: three minutes on a 180 SiC disc, 60 seconds each on a 320 and 600-grit grind, 60 seconds with 1-micron diamond on a red felt cloth, and final polished 60 seconds with 0.05-micron gamma alumina on a flocked twill cloth.

The photomicrographs shown in Figure 4(b) and Figure 4(c) are from the first etch. The ferrite shown in Figure 4(c) is free of disturbed metal and the lamellae in the pearlite is distinct and resolvable and also free of disturbed metal.

Figure 5 is of nodular cast iron prepared in the same specimen holder containing the gray cast specimens as shown in Figure 4. The graphite nodules are flat and intact with no relief polishing.

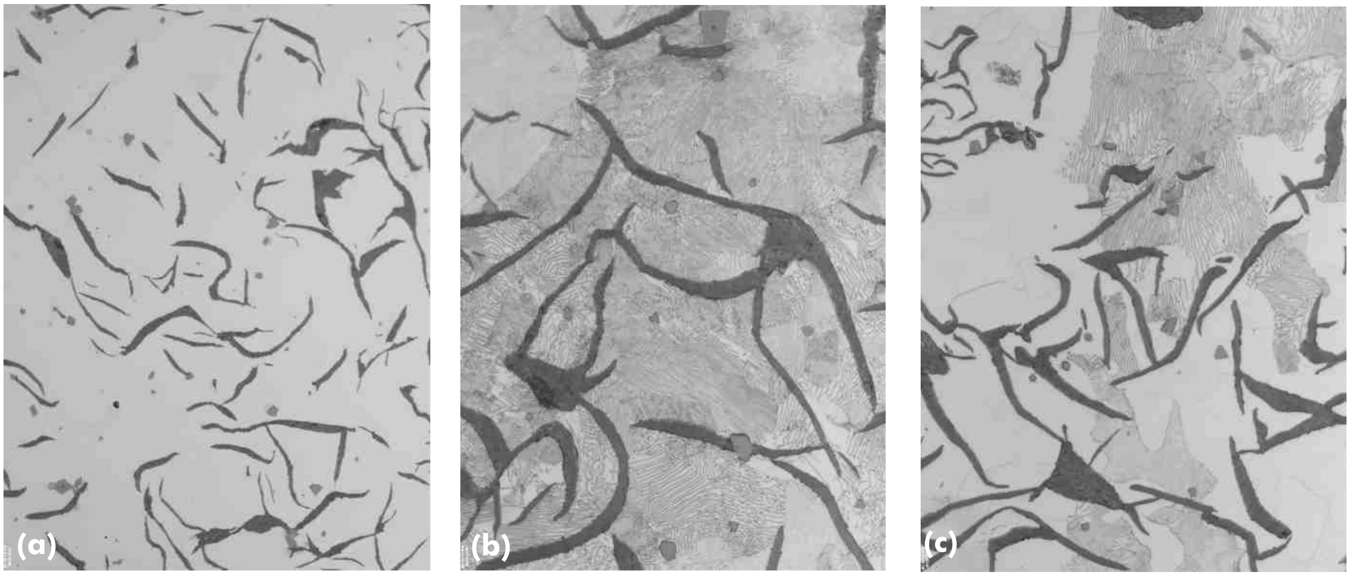

Figure 4: Gray Cast Iron Sample,
(a) 500x, As-Polished, (b) 500x, Etchant: 2% Nital, (c) 500x, Etchant: 2% Nital

Photomicrograph Figure 5(b) is the same field as Figure 5(a) but with polarized light illumination, showing crossed nicols.

The pressure applied to the specimen holder ranges from 18 to 60 psi, and the capability of the automatic polisher to adjust to different pressures during a timed grinding/polishing sequence allows a closer control of the surface finish. The amount of pressure to the specimen holder is evenly distributed to each specimen and prevents some specimens from receiving different degrees of grinding or polishing because of unequal pressures. Also, edge-to-edge flatness is maintained on all specimens. Figure 6 illustrates the surface finish of a carbon steel alloy which has been obtained by manual and automatic procedures. The surface finish shown in Figure 6(a) has been manually ground through a series of decreasing SiC grit sizes starting with 180-grit and followed by 240-, 320-, 400- and 600-grit. The specimen was rotated 90 degrees at each succeeding grinding step and ground until all evidence of the preceding grind was removed. The surface finish shown in Figure 6(b) was automatically ground through 240-grit silicon carbide for 30 seconds then immediately through a 600-grit silicon carbide grind for another 30 seconds. Moreover, only one abrasive disc was used for the 240-grit grind and one abrasive disc for the 600-grit grind to process nine specimens simultaneously.

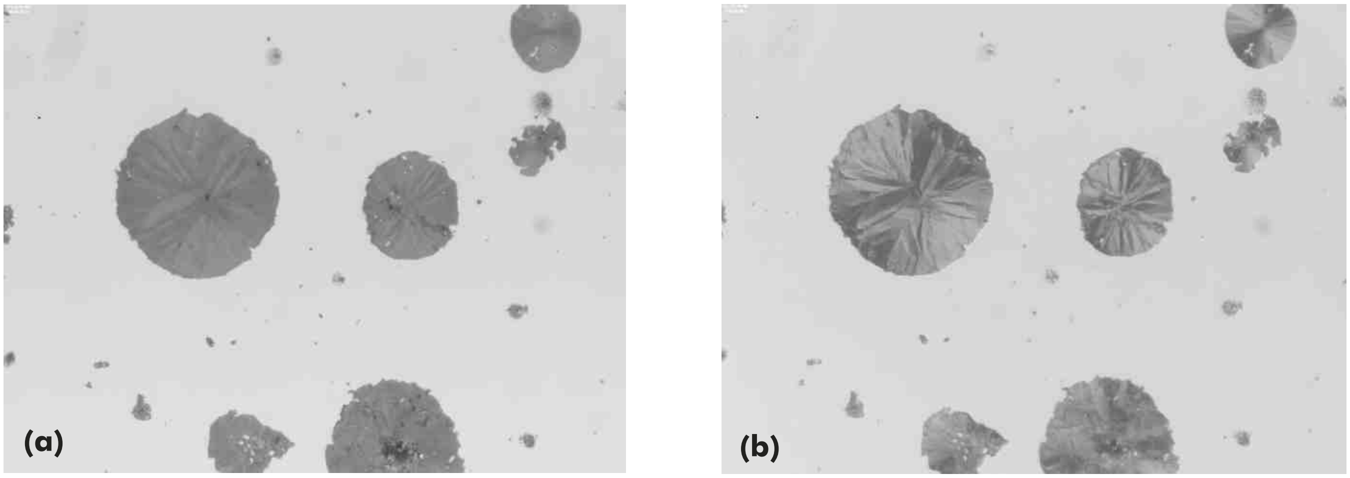

Figure 5: Nodular Cast Iron (a) As-Polished, 500x, (b) As-Polished, under Polarized Light, 500x.

As can be seen, the amount of scratches left in Figure 6(b) is far less than in Figure 6(a) and as a consequence, the time required to remove the 600-grit scratches by a diamond polish would be much shorter. There is no evidence of the prior 240-grit scratches remaining in Figure 6(b) that might be expected if one were to manually grind with a 240-grit and then go directly to a 600-grit grind.

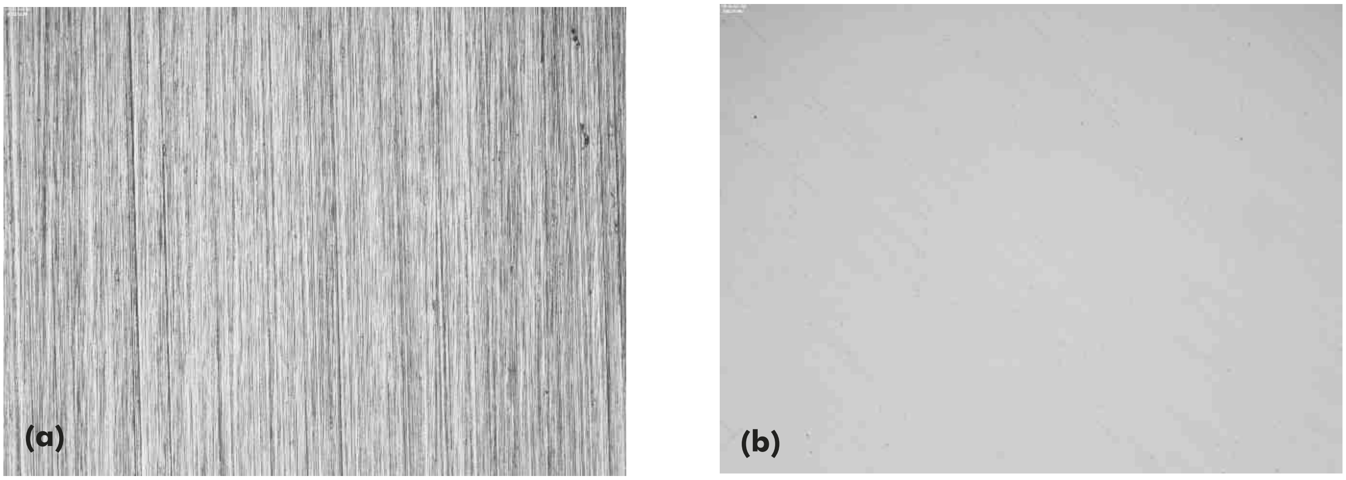

Figure 6: Carbon Steel Alloy Ground by (a) Manual Procedures and (b) Automatic Procedures 100x.

Several experimental studies were conducted using automatic grinding procedures. One, to determine the relative surface roughness of samples after being ground through the various silicon carbide grit sizes compared to samples manually ground through the same grit sizes. Two, the effect of varying pressure on the rate of metal removal.

For the first experimental study, eighteen samples were sectioned from an annealed, $1^1/_4$-inch diameter, austenitic 304 stainless steel was selected because of its homogeneous microstructure. The eighteen samples were divided into three groups, six samples per each group. Samples in Group I were ground manually; samples from Group II were ground automatically using an automatic polisher for 60 seconds at 45 psi, and with a grinding wheel rotation of 300 rpm. Samples from Group III were initially ground in the same manner as those from Group II, then with an additional 30 seconds, 30 psi, and wheel rotation of 200 rpm. All samples in each group started with a 120-grit grind, and as grinding proceeded through the succeeding fine grit sizes, one sample was dropped out after each grind. For example, the sixth samples, which had a 600-grit grind, were ground through all the six grinding steps being evaluated. Each sample from Group I was ground on a fresh, unused paper, and grinding at each step continued until all scratches were uniform and unidirectional. Samples ground automatically were processed six at a time and the scratches were random. Ordinary tap water was used as a coolant, and after each grinding step, the samples were ultrasonically cleaned, rinsed in ethyl alcohol, and dried in a stream of air.

Surface roughness measurements were obtained across the diameter of each sample using an instrument which produced a hard copy of the peaks and valleys of the surface texture. Standard calibration blocks were used to check the sensitivity of the instrument. The results are contained in the table below.

An analysis of the experimental data indicated that, generally, the surfaces were finer on samples from Group II and Group III. Interestingly, there was no apparent difference between the 240- and 320-grit grinds on samples from Group I, but more interesting was the 240-grit grind produced rougher surfaces than the 180-grit grind on samples from Groups II and III. Further analysis of the data obtained on samples from Group II and Group III would seem to indicate that the 240-grit grind could be eliminated without a sacrifice in surface quality, and since there was no difference between the 400- and 600-grit grind, the 400-grit grind could be eliminated, thus having a grinding sequence of 180, 320, and 600.

Comparison of surface roughness between manually and automatically ground samples (millionths of an inch)

SiC GRIT SIZE	GROUP I(b)	GROUP II (C)	GROUP III (d)
120	9	8	7
180	5	3	3
240	4	5	4
320	4	3	2
400	2	1	1
600	2	1	1

(a) Values are AA units

(b) Manually ground

(c) Automatically ground 60 seconds, 300 rpm, 45 psi

(d) Automatically ground same as above, plus an additional 30 seconds, 200 rpm, 30 psi

The second experimental study observed the effect of pressure on the rate of metal removal for an SAE 1045 cold rolled steel and a brass alloy, 85 Cu, 5 Sn, 5 Zn, and 5 Pb.

The effect of pressure on the rate of metal removal is illustrated in Figures 7 and 8. Figure 7 is a graph showing the relationship of the rate of metal removed versus pressure for an SAE 1045 cold rolled steel; Figure 8 is that of a brass alloy (85 Cu, 5 Sn, 5 Zn, 5 Pb).

Nine samples of each alloy were sectioned from a $1^1/_4$-inch diameter rod and both surfaces of each sample surface ground to obtain parallelism. Each sample had a surface area of 1.227 square inches for a total of 11.045 square inches for the nine samples being processed simultaneously. A specimen leveler was used when loading the specimen holder with the unmounted samples to ensure all samples had a common starting plane.

For the studies, certain variables such as time, polishing wheel speed, and water flow were held constant; only pressure was varied. The pressures used were 20, 30, 40, 50, and 60 psi for the steel samples but only through 50 psi for the brass samples. The silicon carbide abrasive grit sizes used were 180, 240, 320, 400, and 600 for both the steel and brass alloys.

After the samples were loaded into the sample holder, thickness measurements were obtained on each sample with hand-held micrometers, then ground for 30 seconds on a 180-grit SiC disc, removed from the automatic polisher, ultrasonically cleaned, and dried. Thickness measurements were taken, then returned to the polisher for an additional 30 seconds of grinding on the same disc after which the sample holder was again removed, ultrasonically cleaned, dried, and thickness measurements obtained. This allowed the amount of metal removed to be ascertained for the first 30 seconds and for the last 30 seconds of grinding. This was done to determine the effective cutting life of an abrasive disc, however, a fresh disc of each grit size was used for each of the pressures evaluated.

The data points plotted on the graphs in Figure 7 and Figure 8 are averages of the nine samples processed simultaneously. As might be expected, more metal was removed from the brass samples than from the steel samples. This is due to the inherent hardness of the two alloys; the SAE 1045 cold rolled steel had an average hardness of 92 HRB and the brass alloy had an average hardness of 65 HRB.

An analysis of the experimental data indicated that generally, the metal removal rates (grinding) obtained with the various grades of silicon carbide using constant speed, water flow and time were relatively high during the first 30 seconds of grinding but decreased rapidly during the last 30 seconds of grinding. This was observed for all the pressures used and is most likely due to the rapid removal of surface asperities initially, then as more of the metal surface came into contact with the abrasive papers, the amount of metal removed decreased rapidly. However, there are other factors that could contribute to a decreased grinding rate. These factors might be (1) dulling or wearing of abrasive particles, (2) dislodging of particles from the paper back, or (3) clogging of the abrasive discs with metal debris. The latter is most likely true for the 400- and 600-grits of silicon carbide.

Further analysis of these data substantiated data from the first experimental studies, i.e., it appeared that several of the grinding steps could be eliminated without a sacrifice in surface quality.

The state-of-the-art of grinding specimens prior to metallographic polishing has not changed much in the last 25 to 30 years. Silicon carbide grinding abrasive has been used in almost every metallographic laboratory throughout the world, either as paper-backed discs or sheets, or as cloth-backed belts. The reason is that silicon carbide is economical and does an excellent job in preparing a specimen for metallographic polishing.

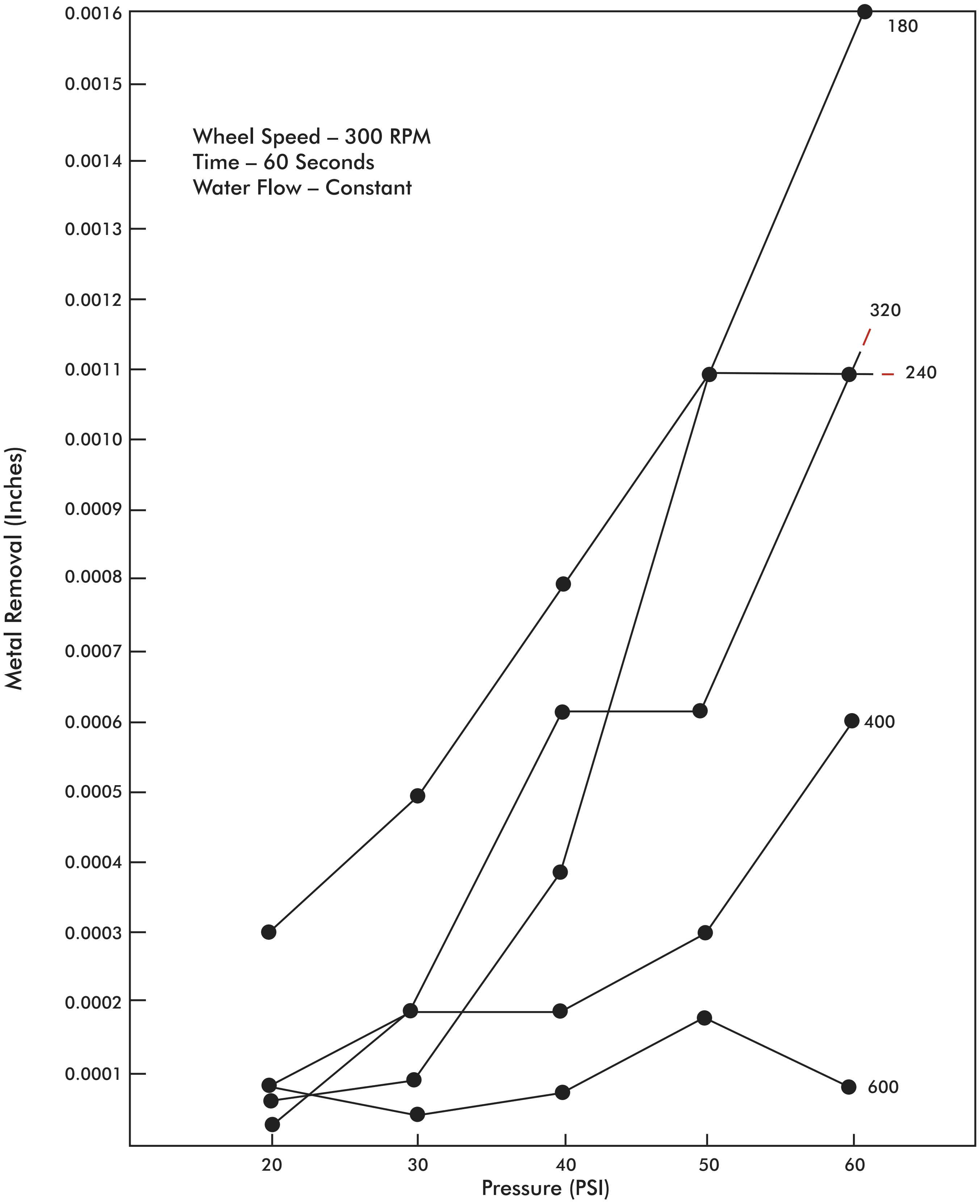

Figure 7: Effect of pressure on the removal of metal on CR1045 steel using silicon carbide abrasive discs

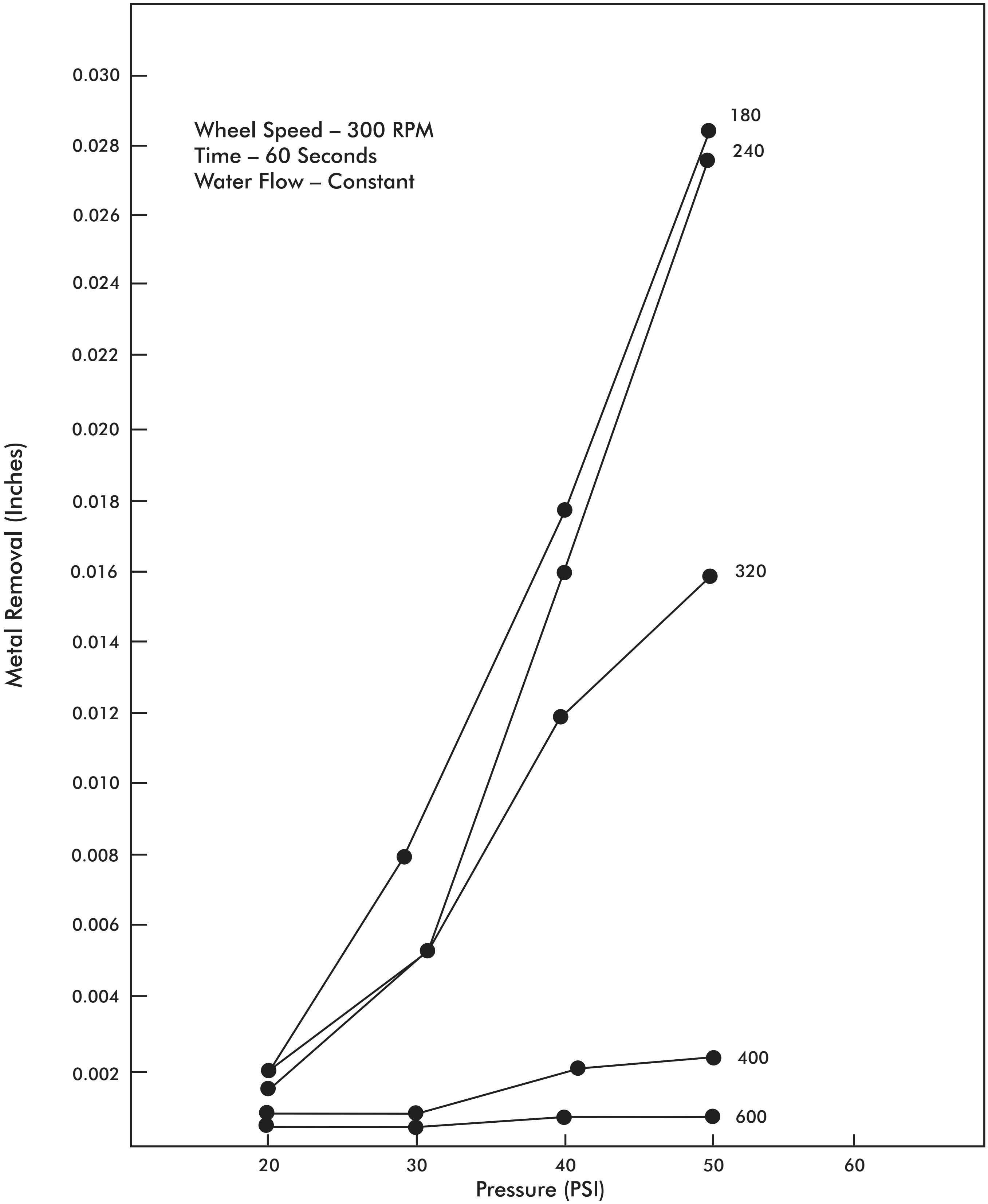

Figure 8: Effect of pressure on the removal of metal on 360 brass using silicon carbide abrasive discs

However, normally it requires grinding with five different, successively finer grades to obtain a surface suitable for polishing. The procedures described herein reduce the original 5-step grind to a 3-step, or in some instances a 2-step grind. This in itself may seem an insignificant change, but when multiplied by the many specimens that are prepared each day in a metallographic laboratory, the saving in time is important because unit costs are reduced and more specimens can be prepared per day.

Until now, the single most important advancement in the preparation of metallographic specimens was the introduction of diamond polishing compounds during the late 1930's or early 1940's. Unfortunately, diamond compounds are the most costly of all consumable items used in the preparation of specimens for microscopic examination. The ability of automatic polishing, where the diamond polishing step can successfully be eliminated, has a tremendous economical impact, and this can only be accomplished where pressure can be controlled very closely, such as with an automatic polisher.

There are basically two types of metallography; quality control or process metallography, and research metallography. The ratio of quality control to research metallography is about seven to one; consequently, quality control laboratories use considerably more of the consumable items, such as grinding papers and polishing compounds or abrasives. Moreover, the volume of specimens processed through a quality control metallographic laboratory is far greater than for a research metallographic laboratory; also the quality of metallographic finish is not so critical as it is for research metallography.

The fact that titanium alloys, nickel base superalloys, and many of the ferrous alloys prepared using the automatic polisher could go directly from a 600-grit silicon carbide grind to a 0.05-micron gamma alumina is due to the drastically reduced deformation that is introduced in the grinding procedures. The depth of deformation is apparently so small that it can successfully be removed by the final polish.

A new era in metallographic practices is introduced. Automatic polishing, capable of varying the pressure applied to the specimen holder with variable speeds not only can eliminate several grinding steps, and in many instances an intermediate polish without a sacrifice in quality, but is also the answer to the quality control manager's problem of keeping pace with increased production demands. Production is increased and the quantity of consumables used is decreased. The challenge of automatic grinding and polishing is there for the far-sighted metallographer to grasp, to use, to experiment, and certainly to expand his expertise.

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LECO Corporation
3000 Lakeview Avenue • St. Joseph, MI 49085-2396 • Phone: 800-292-6141 • Fax: 269-982-8977
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