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AUSTENITIC GRAIN SIZE IN
MARTENSITIC STEELS UNDER
POLARIZED LIGHT

Thesis for the Degree of M. S.
MICHIGAN STATE COLLEGE
Ralph Wenzel Rogers, Jr.
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THESIS

AUSTENITIC GRAIN SIZE IN MARTENSITIC
STEELS UNDER POLARIZED LIGHT

by

RALPH WENZEL ~~ROGERS~~ ROGERS, JR.

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1942

THESE

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Foreward

The original purpose of this investigation was to explore the possibilities of using polarized light to reveal the previous austenite grains in fully hardened steels.

After performing a number of experiments toward this end by mechanically polishing steel specimens, it was decided to use some electrolytically polished specimens to secure an undistorted scratch-free surface.

Because of the incompleteness of the literature descriptive of electrolytic polishing of steels and other metals, it was necessary to investigate and experiment rather thoroughly before satisfactory polishing resulting.

In the end, much of the time devoted to this thesis was spent in the latter phase of the investigation; hence the appendix, which contains the theory, data, and results of the experiments of electrolytic polishing, constitutes a not inconsiderable part of the thesis.

Austenitic Grain Size In Martensitic Steels Under Polarized Light

Throughout the many years of investigation of the metallography of steel, a problem about which there still remains considerable doubt is that of the nature of martensite and the reason for its hardness. Because of the acicular or needle-like structure of martensite and its apparent lack of grain structure, many theories have been evolved on the basis of hypothetical structure in contradiction to the prevalent one that hardening is caused primarily by lattice distortion in the super saturated solid solution of carbon in α -iron, caused by rapid cooling when austenite is severely quenched. For example, Jefferies and Archer¹ formulated their "slip interference" theory on the basis of the formation of extremely small crystals (in martensite) of α -iron--the hardness being caused entirely by the sub-microscopic size of the grains and their resulting resistance to slip.

Heindlhofer and Bain², however, by the application and careful interpretation of x-ray analysis, showed that the crystallograms of martensite are indicative of comparatively large grains. In addition to the x-ray, these investigators used polarized light to show that grains or "blocks" of martensite do exist and are of a size corresponding to that of the parent austenite grains.

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It is the purpose of this paper, then, to establish methods for best revealing the martensite grains clearly, to investigate further the relationship of austenite grains and martensite grains over a range of sizes, and to establish the practicability of using polarized light to determine the grain size of the parent austenite, since the latter is of prime importance in establishing the properties of a steel.

Polarized light is made up of waves whose vibrations are in one plane only as opposed to ordinary light in which the waves vibrate in all directions. Its use in the examination of metals depends on the ability to distinguish between isotropic and anisotropic crystals under polarized light.

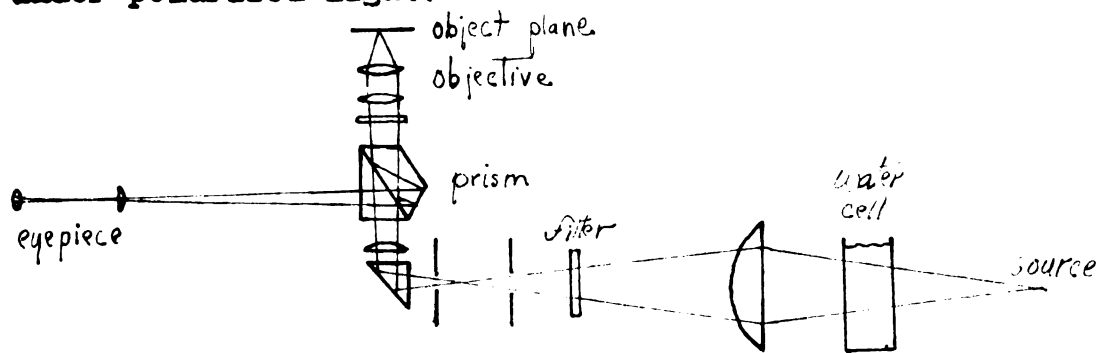


Figure 1. Light Train For Metallograph Equipped With Polarized Light

In the Bausch and Lomb research metallograph used (Figure 1), the light from a tungsten arc is polarized by passing through a Nicol prism in the

the first of these is the fact that the system is not a simple one, but a complex one, in which the various parts are interrelated and interdependent. The second is that the system is not a static one, but a dynamic one, in which the parts are constantly changing and evolving. The third is that the system is not a closed one, but an open one, in which the parts are constantly interacting with the environment. The fourth is that the system is not a linear one, but a non-linear one, in which the parts are constantly interacting with each other in a non-linear fashion. The fifth is that the system is not a deterministic one, but a probabilistic one, in which the parts are constantly interacting with each other in a probabilistic fashion. The sixth is that the system is not a simple one, but a complex one, in which the parts are interrelated and interdependent. The seventh is that the system is not a static one, but a dynamic one, in which the parts are constantly changing and evolving. The eighth is that the system is not a closed one, but an open one, in which the parts are constantly interacting with the environment. The ninth is that the system is not a linear one, but a non-linear one, in which the parts are constantly interacting with each other in a non-linear fashion. The tenth is that the system is not a deterministic one, but a probabilistic one, in which the parts are constantly interacting with each other in a probabilistic fashion.

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vertical illuminator. After reflection from a surface which does not change the polarization of the beam, it passes back into the same prism in such a way that the beam is totally reflected and the effect of "crossed" Nicols is complete darkness. If the polarization of the beam is changed by reflection, the components of the reflected light perpendicular to the original beam are transmitted by the prism and viewed in the eyepiece or camera.

An isotropic crystal is one whose properties are the same in all directions. Polarized light is unchanged on reflection from such a surface so that under "crossed Nicols" total extinction of light occurs in all positions of the crystal.

An anisotropic crystal has different properties in different directions and changes the plane of polarization on reflecting a polarized beam. Hence when viewed under "crossed Nicols", anisotropic crystals will vary in intensity from total extinction to maximum intensity, depending upon the angle formed between each crystal's reflecting plane and the plane of polarization of the beam.

Austenite, which has a face-centered cubic structure, transforms to martensite upon quenching, the latter belonging to the body-centered cubic type. An intermediate product of tetragonal type lattice, which transforms to the body-centered cubic at low temperatures, is believed to form first. If the transformation takes

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place along the crystal planes of the austenite, the resulting martensite should show a grain structure similar to the parent austenite.

As the martensite lattice varies distinctly from an exact body-centered crystal because of the carbon held in super-saturated solution causing the axes to have a ratio as high as 1.06, the martensite grains show anisotropy. If it is composed of crystals of relatively parallel planes, martensite will appear under polarized light to be made up of grains which vary in intensity according to their orientation. Moreover, on revolving the martensite specimen, the intensities will change, each grain going through two maxima and two minima in a 360° revolution.

Some of the practical difficulties associated with polarized light make its use limited to a greater extent than is at first apparent. For example, the low intensity and low contrast of the reflected light make photography difficult. Also much that can be seen visually by rotating the specimen and changing the intensities of the grains cannot be retained on a photomicrograph. The amorphous or "Beilby" layer of flowed metal formed on polishing is isotropic and must be removed to form a reflecting surface of the anisotropic martensite only. This is usually accomplished by etching, but the resulting roughening of the surface makes the intensity of the reflected beam considerably less.

Methods For Revealing Martensite Grains

The technique used to secure a surface for investigation under polarized light is important because of the rather exacting limits set by its use. Various factors were investigated in order to determine the optimum conditions for revealing the grain structure of martensite under polarized light. Particular attention was devoted to the small-grained specimens since the large-grained martensite can be revealed with comparative ease by any methods satisfactory for the former.

The factors investigated were the methods of removing the amorphous layer of disturbed metal and the effect of tempering. The methods used to remove the surface metal were etching and electrolytic polishing.

As mentioned before, it is necessary to remove the disturbed layer of metal, which is amorphous and isotropic in nature, in order to obtain reflection from the anisotropic metal beneath. If this is done by etching, it is necessary to avoid over-etching since the already low intensity of reflected polarized light is rendered considerably lower by roughness caused by too deep an attack.

The ordinary etching solutions used did not seem to improve the revelation of a small grain size except for a special reagent for austenite grain boundaries

in martensite. This reagent, proposed by J.R. Vilella⁶, consists of one gram of picric acid in five parts of hydrochloric acid and 95 parts of ethyl alcohol. In large-grained martensite, the previous austenite grains show up under ordinary light with this reagent (figure 2), but small grained specimens show little or nothing. With small grains under polarized light, fair results can be obtained with this reagent. Much of its advantage lies in its slow rate of attack so that the degree of etch can be closely controlled and over-etching avoided.

The use of electrolytic polishing to remove the surface layer offers the advantages of a strain-free, undisturbed surface secured by electropolishing instead of mechanical polishing to remove scratches left by the fine emery paper. Here the danger of roughening the surface and diffusing the light is again present and conditions for obtaining a good polish were investigated to considerable length as the literature on this process is quite incomplete and misleading. The theory, results and data of this part of the investigation are contained in an appendix to this thesis.

In general, as might be expected, it was found that with electropolishing no further etching is needed. Sometimes, however, a light attack by Vilella's reagent, 2% picral, or 2% nital was found to increase

the clarity of the picture. It was found best to examine the specimen unetched and then to etch if necessary as no definite procedure could be specified.

Figure 3 is a specimen of S.A.E. 1090 steel quenched from 1550° F., electropolished and not etched, while Figure 4 is the same steel etched with Vilella's reagent. Figure 5 is S.A.E. 1090 steel, quenched from 1700° F., electropolished and not etched. Figure 6 is S.A.E. 1090 steel quenched from 1900° F. and etched with nital.

Figures 7,8,9 and 10 are S.A.E. 1040 steel in the annealed, normalized, water-quenched, and oil-quenched conditions under polarized light after electropolishing and etching with nital.

The purpose of tempering martensite steels for observation under polarized light is to complete the transformation from tetragonal to more nearly cubic body centered crystals. Probably because the latter is in a more strain-free state, the crystal planes are more perfect and so the light is reflected in a more orderly manner with resulting clarity in the pictures.

Figures 11,12,13 are specimens of S.A.E. 1090 steel quenched from 1450° F. Figure 11 shows the resulting structure while figures 13 and 12 were tempered at 450° F. for 15 minutes and 30 minutes respectively.

Figures 14 and 15 are photomicrographs of a specimen of S.A.E. 1090 steel quenched from 1900° F., before and after tempering at 475° F. for 15 minutes.

From these photomicrographs it is seen that tempering is only a slight aid in improving the clarity of small grained martensite under polarized light, but it improves the observation of large grained steel to a greater degree.

Figure 2

SAE 1090
quenched from 1900° F.
etched with Vilella's
reagent
100x

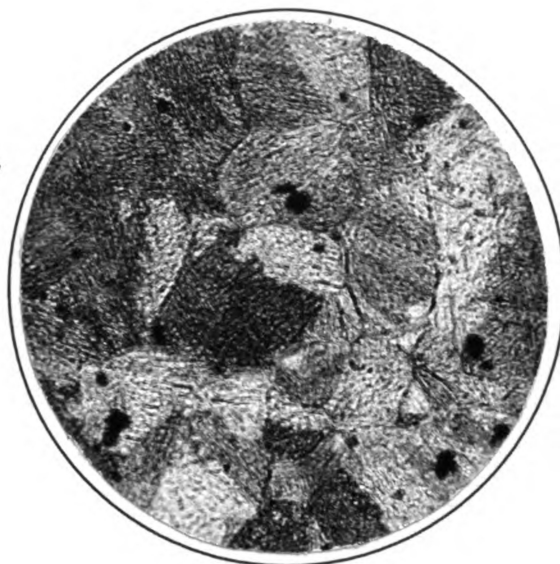
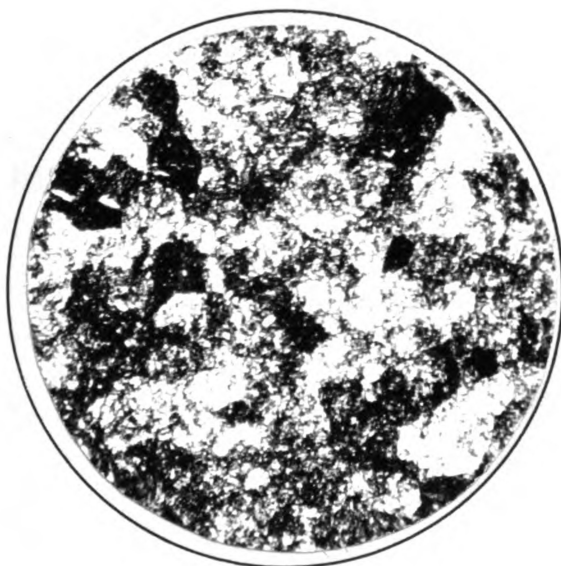


Figure 3

SAE 1090
quenched from 1550° F.
tempered at 450° F.
electrolytically pol-
ished, no etch
100x
polarized light



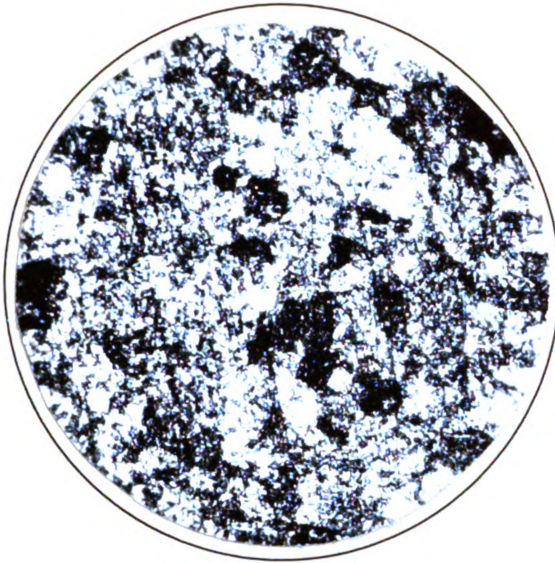


Figure 4

SAE 1090

quenched from 1550° F.

tempered at 450° F.

electrolytically polish-

ed and etched with

Vilella's reagent

100x

polarized light

Figure 5

quenched from 1700° F.

tempered at 450° F.

electrolytically pol-

ished, no etch

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polarized light

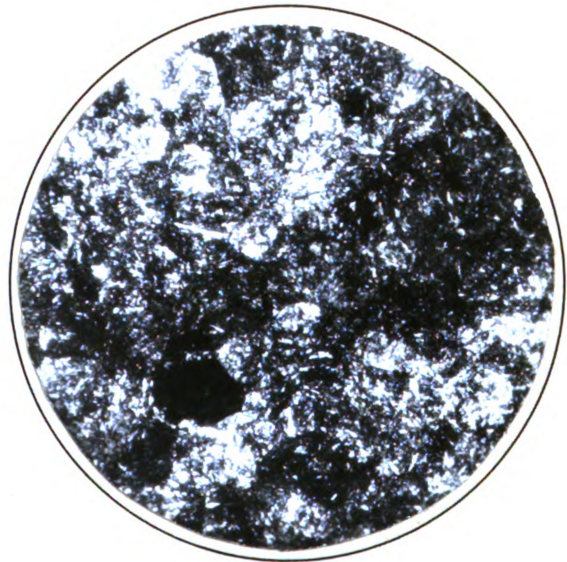




Figure 6

SAE 1090

quenched from 1900° F.

electrolytically pol-

ished and etched with

nital

375x

polarized light

Figure 7

SAE 1040

annealed

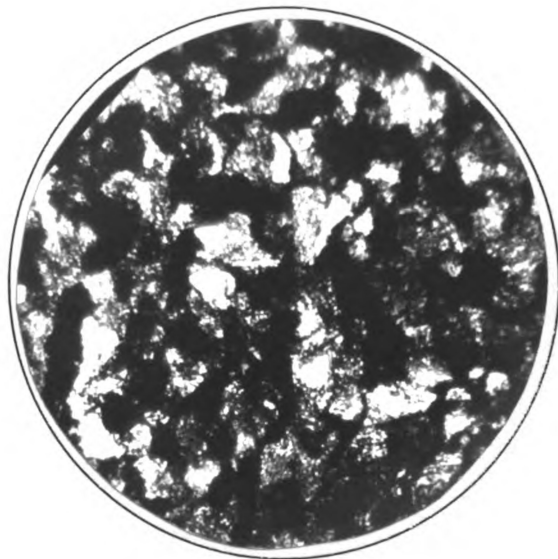
electrolytically pol-

ished and etched with

nital

375x

polarized light



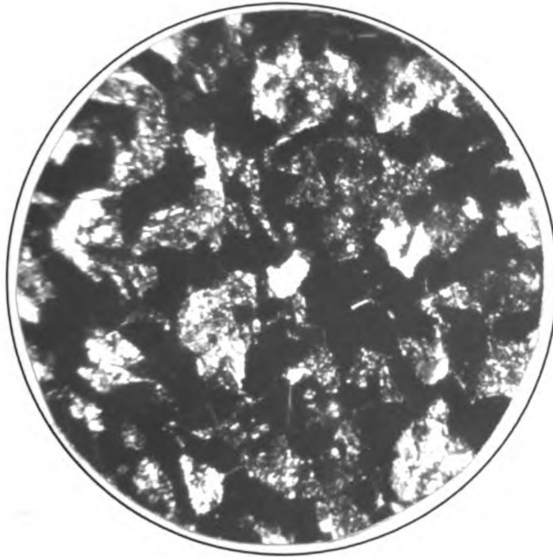
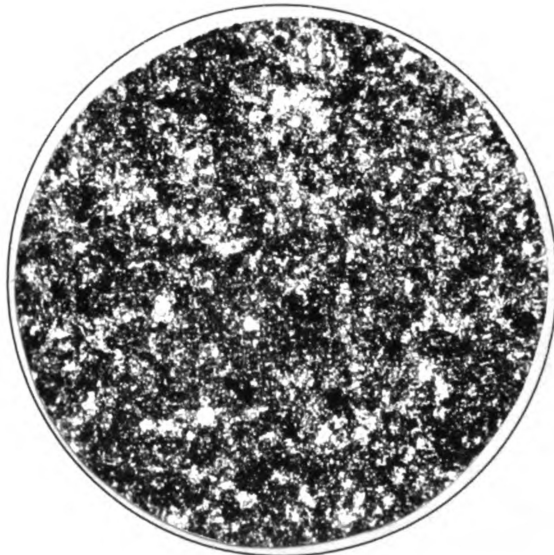


Figure 8
SAE 1040
normalized
electrolytically pol-
ished and etched with
nital
750x
polarized light

Figure 9
SAE 1040
water quenched
electrolytically pol-
ished and etched with
Vilella's reagent
100x
polarized light



(13)

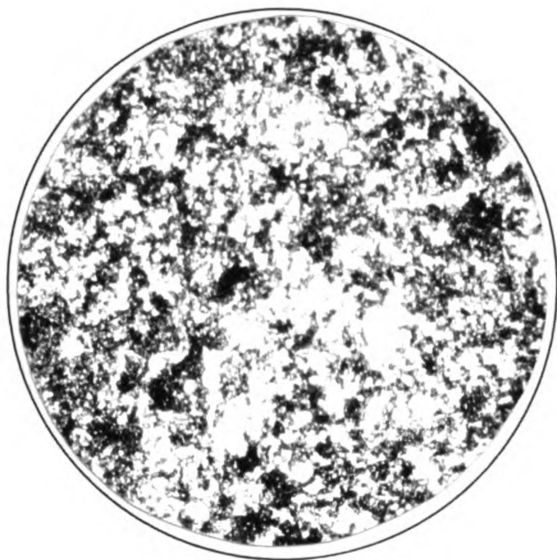


Figure 10

SAE 1040

oil quenched

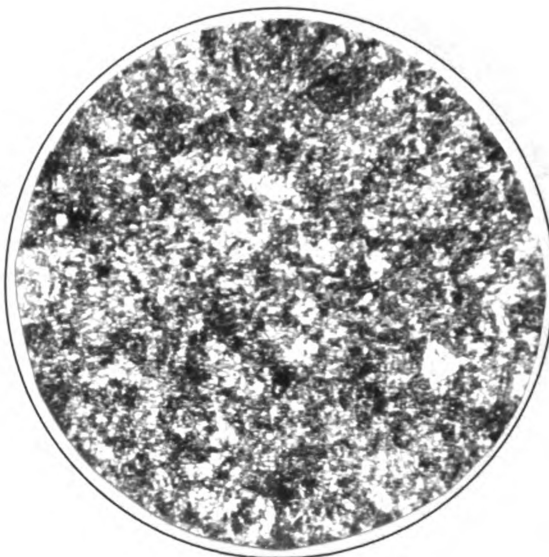
electrolytically polished and etched with

picral

100x

polarized light

Figure 11
SAE 1090
quenched from 1450° F.
etched with
Vilella's reagent
100x
polarized light



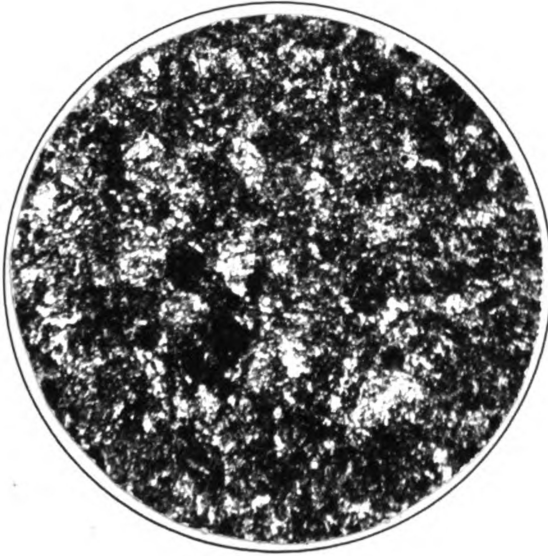


Figure 12
SAE 1090
quenched from 1450° F.
tempered for 30 minutes
at 475° F.
etched with
Vilella's reagent
100x
polarized light

Figure 13
SAE 1090
quenched from 1450° F.
tempered for 15 minutes
at 475° F.
etched with
Vilella's reagent
100x
polarized light

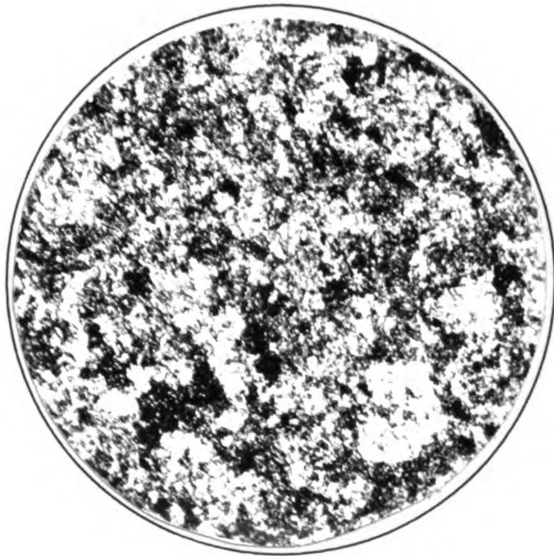
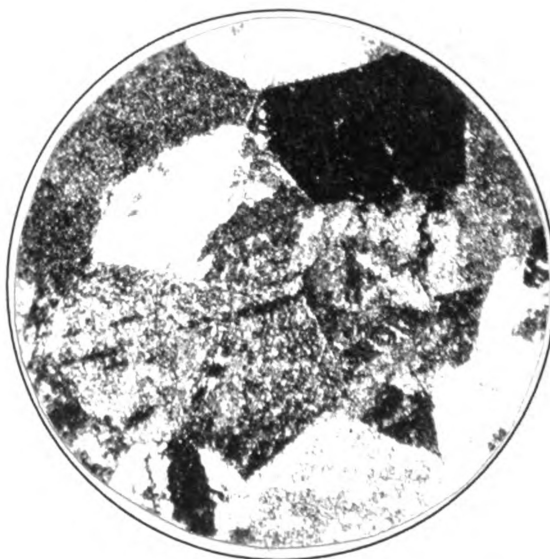




Figure 14
SAE 1090
quenched from 1900° F.
etched with
picral and nital
100x
polarized light

Figure 15
SAE 1090
quenched from 1900° F.
tempered 15 minutes
at 475° F.
etched with
Vilella's reagent
100x
polarized light



Austenite Grains and Martensite Grains

There are a number of methods used by the metallurgist to determine the previous austenitic grain size in martensitic steels. Among these are the arrested quench, gradient quench, slow cooling, preferential oxidation, heat tinting and case carburization methods and the use of special reagents. All of these are limited to steels of certain carbon ranges or by the necessity of using unusual equipment, or because of the inordinate length of time required for the test. Martensitic steels of approximately eutectoid composition are those in which it is most difficult to establish the austenitic grain size so that for these experiments S.A.E. 1090 steel was used. Eutectoid steel also has the advantage of consisting of only one constituent-martensite- in a quenched structure, so that there is no other structure present to complicate the observations.

To establish the relationship between austenite grains and martensite grains, samples of steel were quenched from 1850° F. and 1450° F. into cold water. Samples from the same bars were submitted to a gradient quench from the same temperatures. This consists of quenching one end of the specimen only--so that at a certain point along the axis of the specimen, the cooling rate is such to cause the formation of troostite

in the grain boundaries of the austenite as rapid cooling occurs. At room temperature this zone consists of martensite outlined by thin lines of dark-etching troostite, thus showing the location of the previous austenite grain boundaries.

Figures 16 and 17 were quenched from 1450° F., the former a gradient quench under ordinary light and the latter an identical steel after regular quenching and photographed under polarized light.

Figures 18 and 19 represent a similar treatment from 1850° F., the former being the gradient quench.

From these comparisons, it is readily seen that the grains in martensite under polarized light are of the same order of magnitude as the parent austenite grains. Also, the difference in clarity of the photomicrographs by both methods between the large and small grains is illustrated. The temperatures of 1450° F. and 1850° F. were chosen to secure austenite grains before and after the coarsening temperature of this steel was reached, and so a considerable difference in the austenitic grain size obtains upon quenching.

Figures 20-27 together with Figures 17 and 19 show the structure of martensite under polarized light secured by quenching steel from temperatures which varied by increments of 50° F. between 1450° F. and 1900° F. From a comparison of these pictures, it is

seen that the specimens quenched from 1700° F. and below are small grained and lacking in distinctness, while those quenched from above this temperature are both large grained and well defined. The border case, Figure 24, shows evidence of duplex structure but lacks the distinctness of Figures 25 and 26. The coarsening temperature of this steel may be established at about 1750° F. by these photomicrographs. That more than merely increased grain growth occurs at this temperature seems likely since such a considerable difference in appearance and distinctness results from quenching from above and below this temperature.

The general appearance of the grain revealed by polarized light is typical of a face-centered crystal such as austenite--the grain being equi-axed and roughly hexagonal, especially in the large-grained specimens. In examining martensite under polarized light, care must be taken in determining the size of grains. For example in Figure 28, several orientations within one grain are visible as shown by the same picture under ordinary light, Figure 29. By rotating the specimens, the areas of a grain showing different orientations will display a maxima or minima at approximately or exactly the same angle. Figure 30 illustrates this, as it was taken at an angle of 33 degrees from Figure 5.

This is the same way that "twinned" austenite grains show different orientations under polarized light and for the same reason; stresses are relieved

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in a grain by a "twisting" of a part of the structure so that the orientation varies in different parts of the original grain. As the light reflected varies in intensity according to the angle made with the plane of polarization, the differently oriented parts vary in intensity. Twinning is especially evident in Figures 27 and 6.

Magnifications beyond 100 diameters showed no additional detail under polarized light and are at a disadvantage as the number of grains under observation is correspondingly smaller. Since the percentage of well-outlined grains is low, to determine the size of the grains, as large a field as is possible should be used. Figures 6, 7, and 8 and Figure 31, which is of the same area shown in Figure 24, were taken at magnifications other than 100 diameters.

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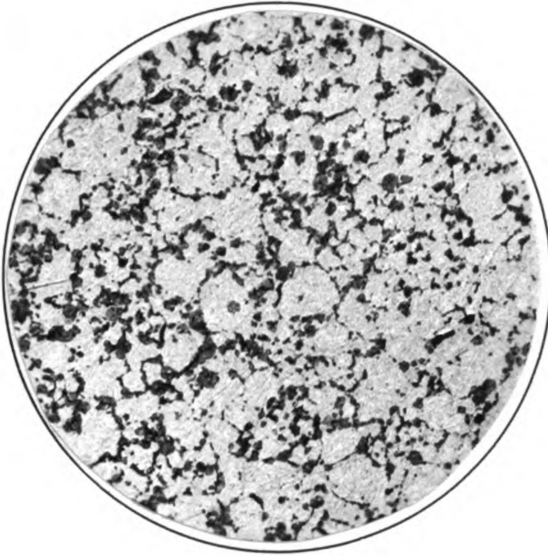


Figure 16
SAE 1090
gradient quench from
1450° F.
100x

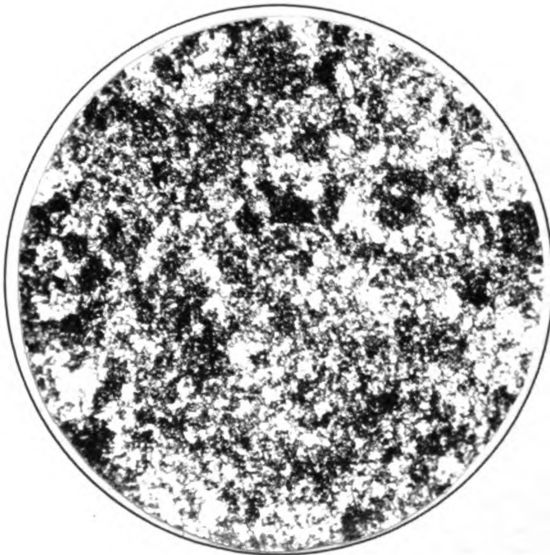


Figure 17
SAE 1090
quenched from 1450° F.
tempered at 400° F.
100x
polarized light

Figure 18
SAE 1090
gradient quench from
1850° F.
100x

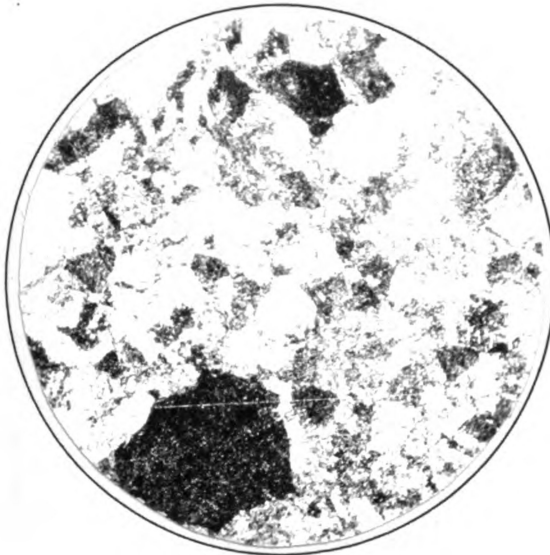
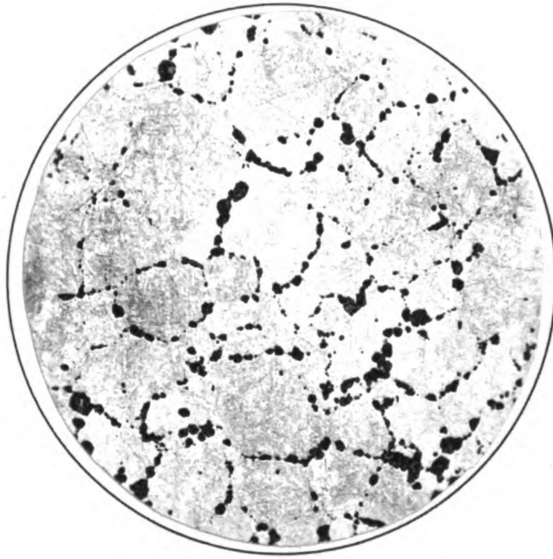


Figure 19
SAE 1090
quenched from 1850° F.
tempered at 400° F.
100x
polarized light

Figure 20
SAE 1090
quenched from 1500°F.
tempered at 450° F.
100x
polarized light

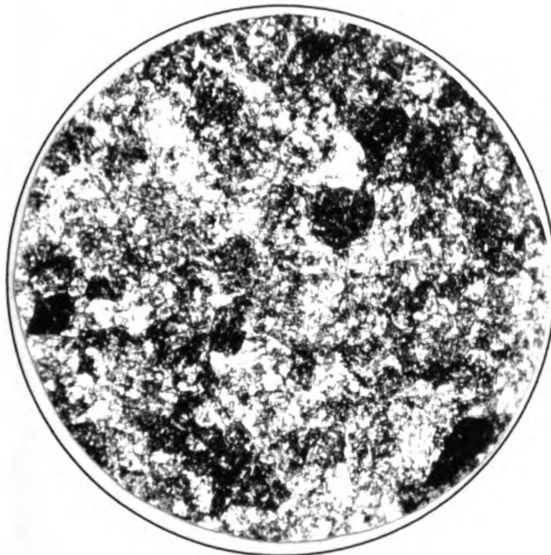
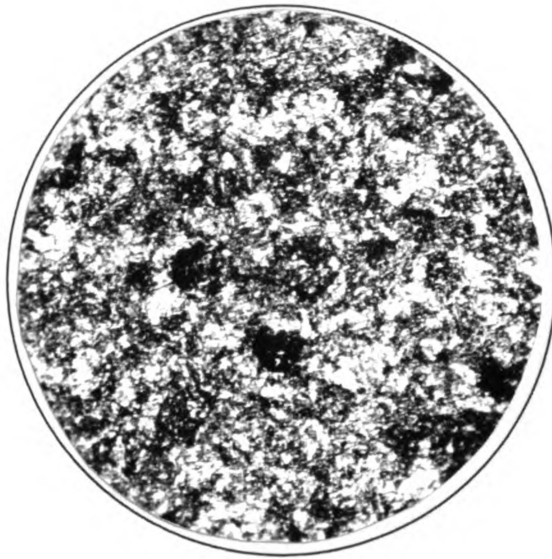


Figure 21
SAE 1090
quenched from 1550° F.
tempered at 450° F.
100x
polarized light

Figure 22
SAE 1090
quenched from 1600° F.
tempered at 450° F.
100x
polarized light

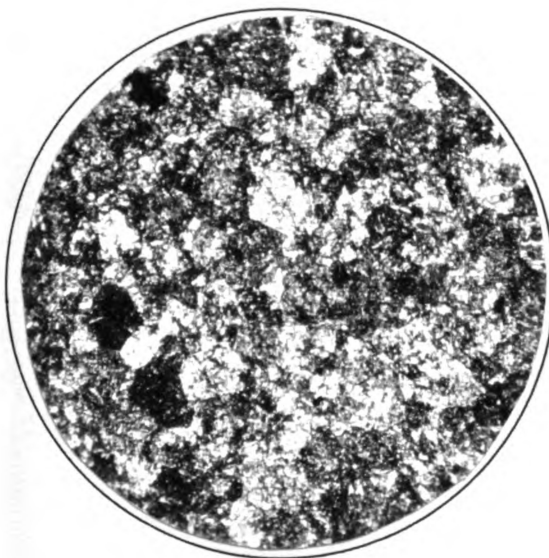
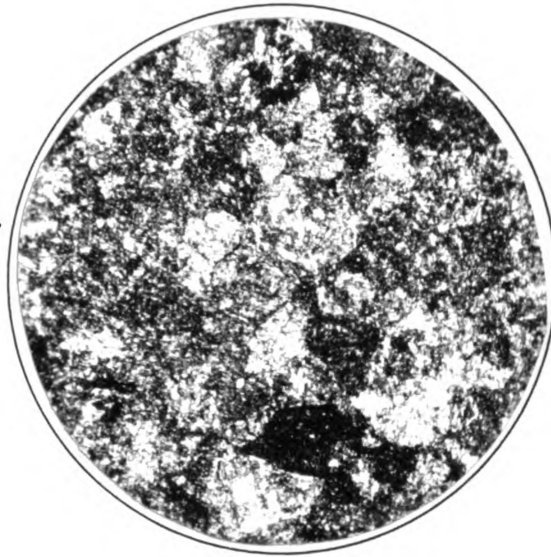


Figure 23
SAE 1090
quenched from 1650° F.
tempered at 450° F.
100x
polarized light

Figure 24
SAE 1090
quenched from 1700° F.
tempered at 450° F.
100x
polarized light

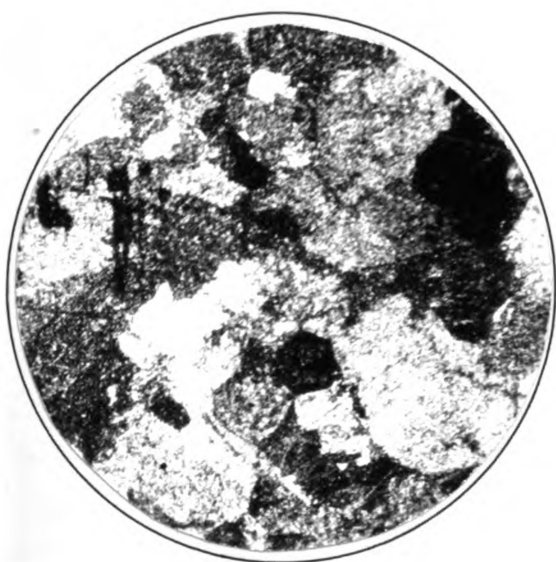
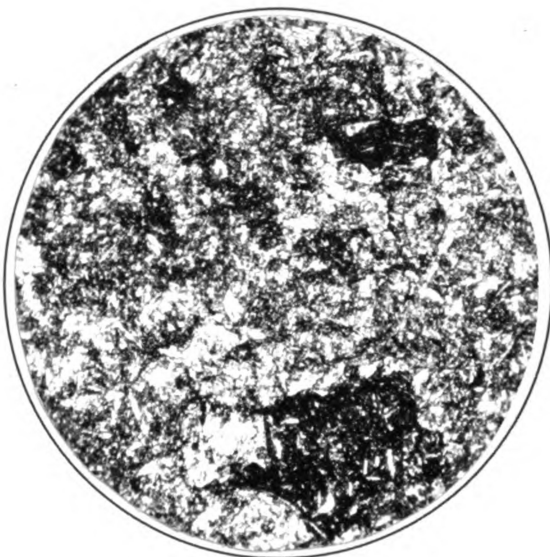


Figure 25
SAE 1090
quenched from 1750° F.
tempered at 450° F.
100x
polarized light

Figure 26

SAE 1090

quenched from 1800°F.
tempered at 450° F.

100x

polarized light

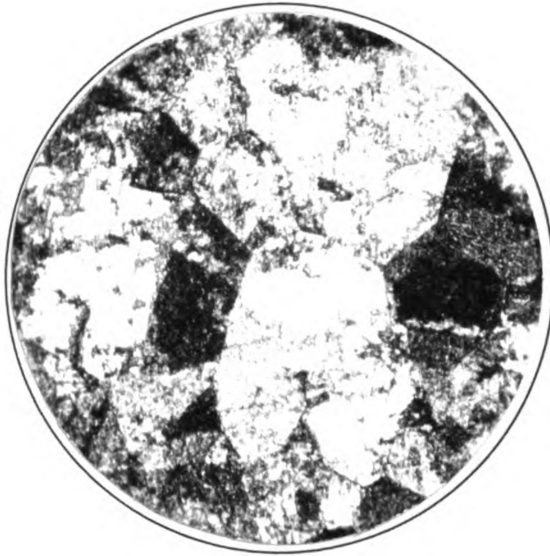


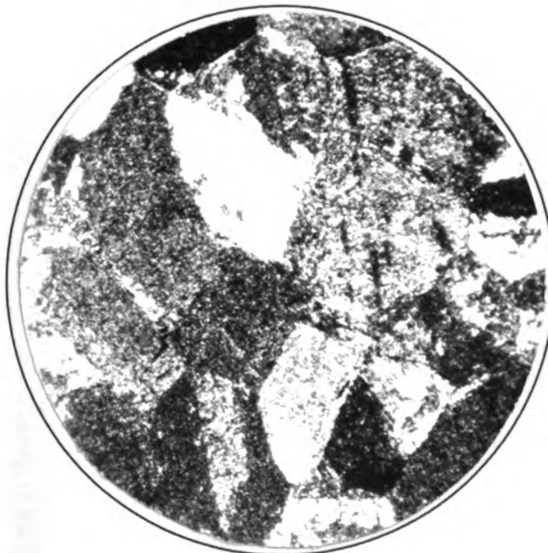
Figure 27

SAE 1090

quenched from 1900° F.
tempered at 450° F.

100x

polarized light



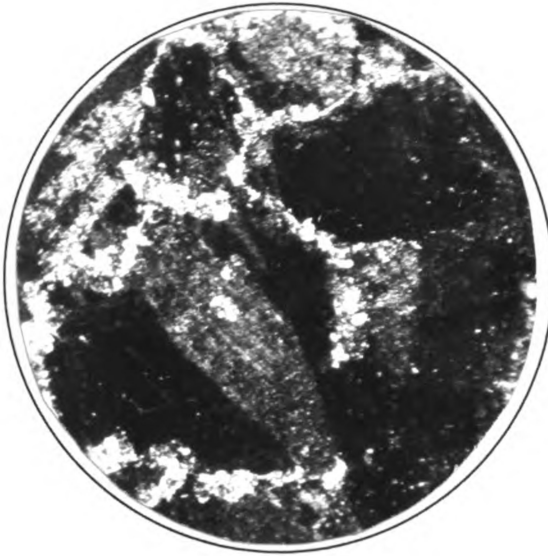
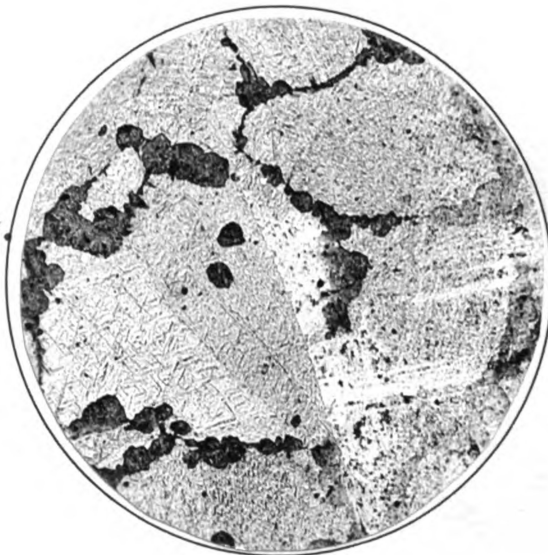


Figure 28
SAE 1090
quenched from 1900° F.
etched with
Vilella's reagent
100x
polarized light

Figure 29
SAE 1090
quenched from 1900° F.
etched with
Vilella's reagent
100x



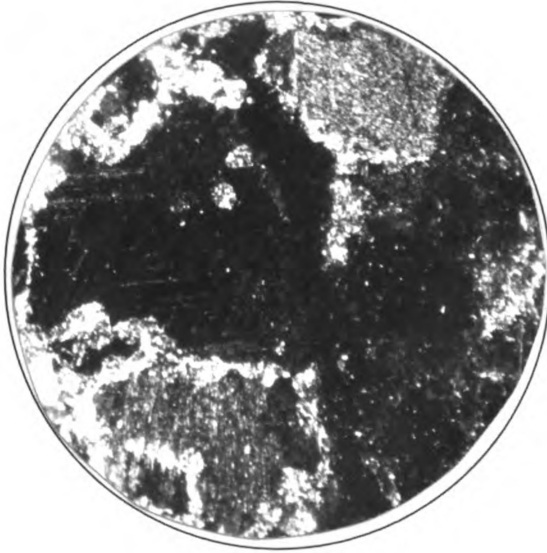
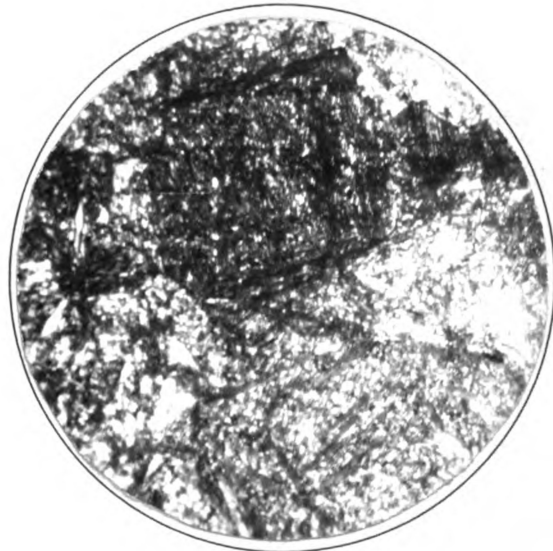


Figure 30
SAE 1090
quenched from 1900° F.
etched with
Vilella's reagent
100x
polarized light

Figure 31
SAE 1090
quenched from 1700° F.
electrolytically
polished
500x
polarized light



Summary

From the series of pictures taken under polarized light, it may be seen that the size of grains revealed is of the same order of magnitude as the parent austenite. According to Heindlhofer and Bain², these are actual grains of martensite, and not merely traces of the austenite.

For the purposes of making an estimate of the grain size of the parent austenite and for a determination of the coarsening temperature of a steel, polarized light can be used successfully. For steels near the eutectoid composition, this method is especially valuable and is the only simple method available. In other steels, it offers advantages in time saving as no slow annealing is required; quenching and a fifteen minute tempering is the only heat treatment required. In steels which are already hardened, this is the only method for ascertaining the grain size and structure, except for special martensitic etches, the successful use of which is frequently impossible.

For further investigation, it is suggested that the alloy and plain carbon steels over a range of compositions be studied to determine the limits of polarized light for as the composition of martensite approaches alpha-ferrite, the anisotropy disappears

at some point. This was borne out by attempts to observe 1010 steel under polarized light which were unsuccessful.

Appendix

APPENDIX

Electrolytic Polishing of Steel

The electrolytic polishing of metals was first attempted by Jacquet who successfully polished copper^{3,5} and tin⁷. by this method. Briefly, it consists of the reverse of electro-plating; the specimen is placed at the anode of an electro-plating bath and conditions are so controlled as to secure the removal of a layer of metal at the anode rather than an even deposit at the cathode.

Conditions for successful electrolytic polishing must be controlled very carefully to avoid a rough surface caused by pitting, etching, or uneven attack. The variables which have an influence on the quality of polish are the electrolyte, current density, voltage, time, agitation, temperature, and the shape, area and location of the cathode.

These variables have been the subject of considerable investigation and while results have been obtained for many metals, there is still lacking enough information to be able to duplicate results in most instances.

It was the object of this investigation to determine the conditions necessary and to construct the apparatus with which to electrolytically polish commercial steels quickly and so secure a strain-free, scratchless surface.

A procedure for systematic investigation was also devised as a guide for future investigations of this type of work.

The most important factor in either polishing or plating is the proper choice of electrolyte. Investigators agree that a viscous, non-conducting film is necessary to establish a concentration gradient between the elevations and depressions of the surface to be polished. Uhlig⁴ points out that according to theoretical electricity, high gradients of potential exist in the vicinity of points of great curvature-- even though the total gradient be small--hence at the elevations a better anode efficiency exists and the removal of metal proceeds at a greater rate than in the depressions.

This film may be an oxide film, a layer of gas, insoluble products of electrolyte and metal, a static liquid film or any combination of these, and it is especially necessary to secure a good film for the polishing of non-homogeneous alloys to avoid uneven attack on different phases⁸.

The choice of electrolyte then should be one in which such a film is formed. The formation of metal salts which are but slightly soluble in the electrolyte satisfies the requirements. The addition of organic liquids to reduce the diffusion of the film and to lower the conductivity of the electrolyte is recommended by Uhlig⁴.



To determine the proper current density and voltage, and whether the electrolyte will result in satisfactory polishing, a curve of current versus voltage should be made, using the metal and electrolyte in question. The curve for a successful combination will have a shape similar to one of the following, which were secured by various investigators.

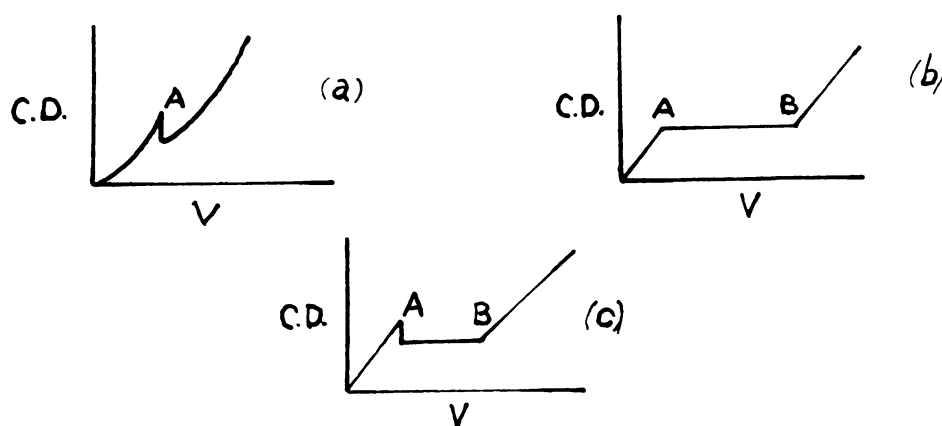


Figure 32. Voltage-Current Density Curves

For curves b and c of Figure 32, the range for successful polishing occurs in the flat part between A and B^{9,10,5}. In the portion of the curve above B, gas is evolved and an uneven surface results while below A, etching occurs⁵. For curve a, the critical point A is the lower limit at which polishing proceeds successfully. Below this, the rate of diffusion of the film exceeds its rate of formation and hence the film is **not** maintained⁴.

Experimental polishes at the voltage values so determined will ascertain their correctness, and by varying conditions such as temperature, cathode distance,

and the like, the optimum conditions for polishing the metal can be determined.

Apparatus

The apparatus used for electrolytic polishing was based upon the use of 220 volt direct current which was available in the laboratory. Since the voltages to be applied were not that high, the following setup was used to secure the desired voltage and current.

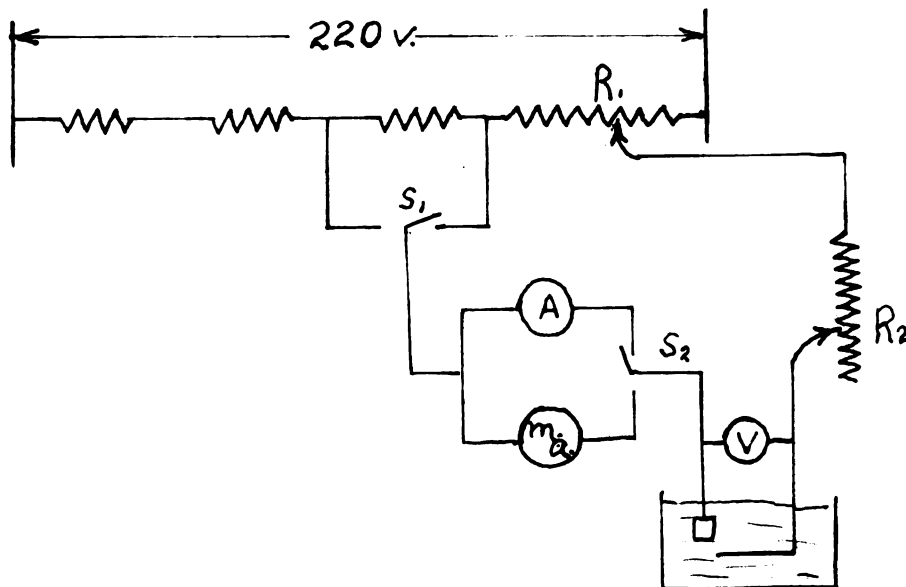


Figure 33a. Electrical Circuit for Electrolytic Polishing

The switch S_1 permits the use of high or low voltage while S_2 throws in either the ammeter or

milliammeter. The rheostats R_1 and R_2 control the current, the latter being used for close control.

The cell consisted of a battery jar, usually set in cooling baths of ice water, with a motor driven stirrer. The cathode was placed parallel to the bottom while the specimen was clamped to the holder. This holder was built from an old microscope frame and makes possible close regulation of the depth of the specimen in the electrolyte, and easy removal of the specimen from both, because of a hinged frame. Figure 33b is a photograph of the cell, including the holder and stirrer, but without the external cooling bath. Figure 33c shows the control panel as diagrammed in Figure 33a.

(35)

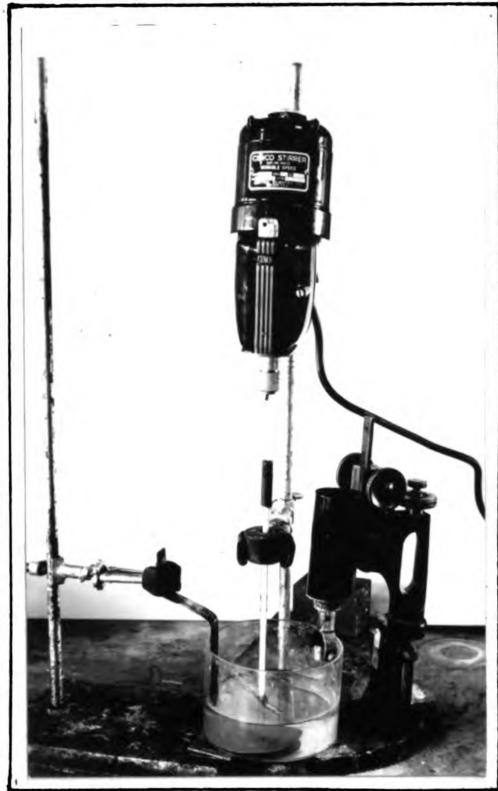


Figure 33b. Electrolytic Polishing Apparatus

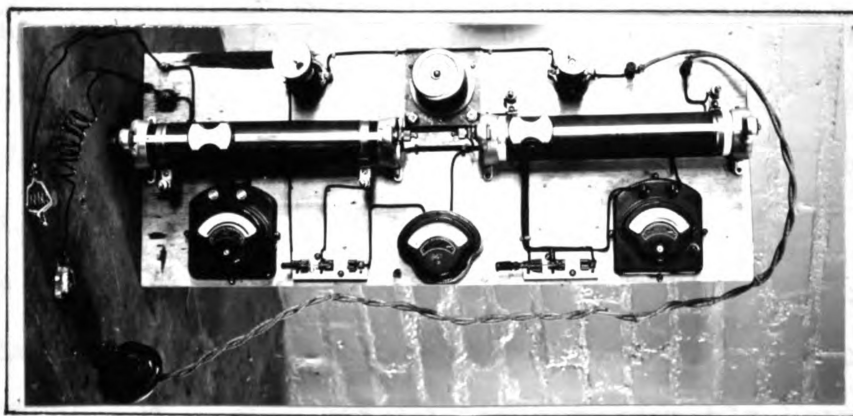


Figure 33c. Control Board

Experiments on Electrolytic Polishing

The experiments on electrolytic polishing of steel were primarily concerned with S.A.E. 1090 steel since that is what is used throughout the polarized light experiments. Other steels were also polished electrolytically to verify the conclusions reached with 1090. These included S.A.E. 1040, 1010, and 10120.

For the first attempts to polish electrolytically, a storage battery was used for the current source. After a number of trials, the voltage secured proved to be too low to polish steel so that the 220 volt D.C. setup previously described was devised and proved successful.

The electrolyte used in all of the experiments was one recommended for steel by a number of writers and consisted of 765 parts of acetic anhydride, 185 parts of perchloric acid, and 50 parts of water. The film formed in this electrolyte with steel is said to be $[\text{Fe}_3\text{Ac}_6(\text{OH})_2]\text{ClO}_4 \cdot 4\text{H}_2\text{O}$ by Jacquet and Rocquet¹¹. The solution is made up by chilling the chemicals before and during mixing, as considerable heat is evolved and the danger of a violent reaction is present. The solution is allowed to stand for 24 hours before using.

Most reports on electrolytic polishing emphasize the current density as the prime factor in securing a



successful polish, so that in the first of these experiments the current density was the primary subject of investigation. However, this did not prove to be very successful in controlling the quality of polish because of the difficulty encountered in keeping a constant area in contact with the electrolyte. As the apparatus was designed to be a simple and rapid means of polishing metals, the sample to be polished was simply suspended in the electrolyte, the total area immersed being subject to polishing action. Hence, when the solution was agitated, the wave motion caused a variation in area. Also, the depth of immersion was difficult to ascertain because of the low surface tension of the solution showing in the high degree of adhesion to the metal. Coating the sides of the specimen with paraffin was tried in order to maintain a constant area but the paraffin was attacked by the electrolyte and failed to protect the specimen. For these reasons, control by current density was not consistent and results were not always successful.

Controlling the polishing by means of the applied voltage was next attempted. Since the voltage does not vary with the area in contact with the electrolyte, the above difficulties were eliminated. To determine what voltage values to use, the voltage-current curves were secured for each metal. The technique employed

(38)

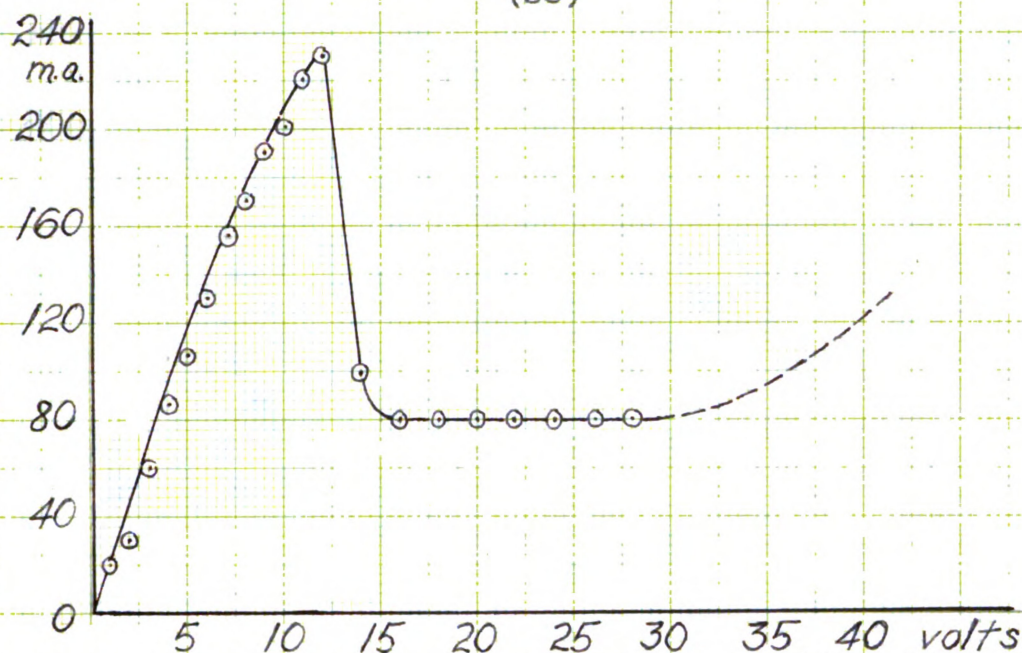


Figure 34. Voltage-Current Curve of 1010 Steel
Current values are proportional to area

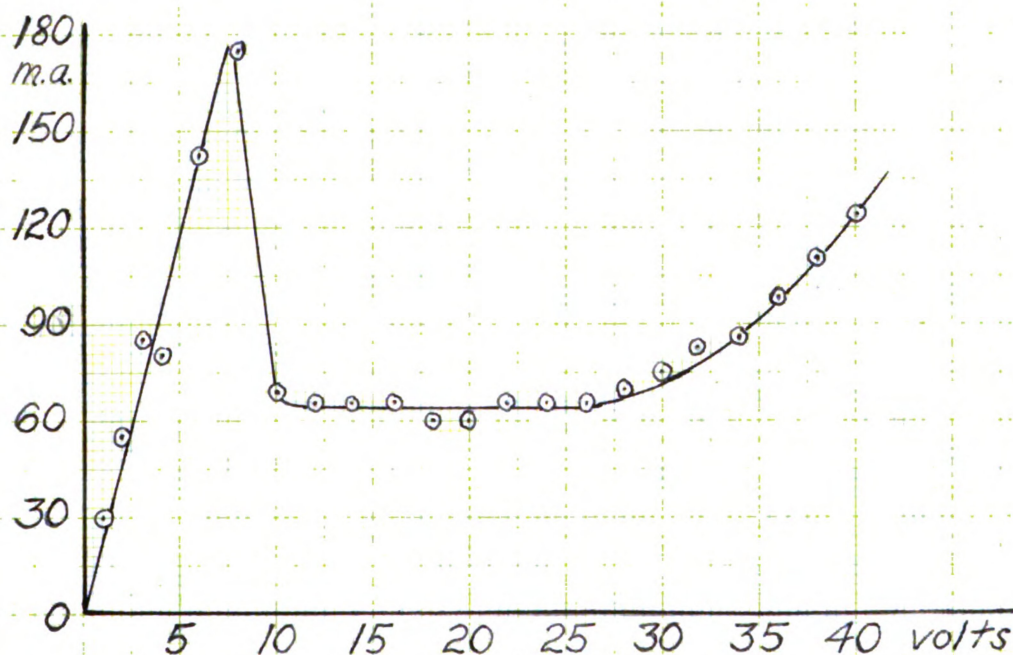


Figure 35. Voltage-Current Curve of 1040 Steel

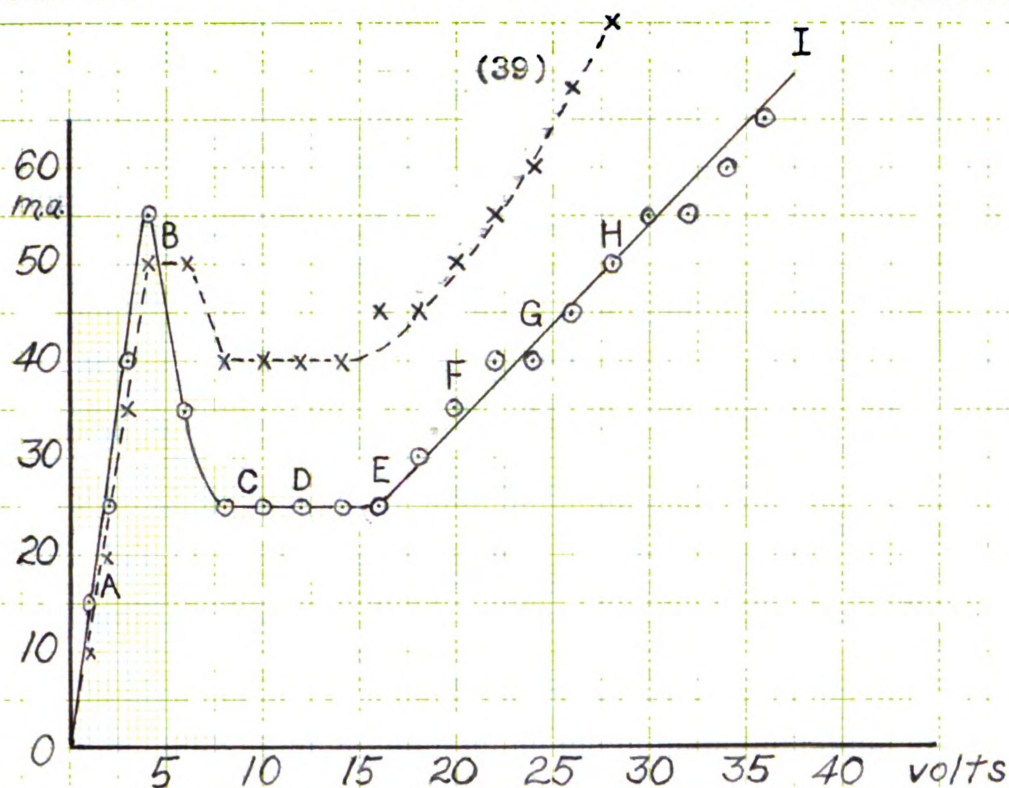


Figure 36. Voltage-Current Curve of 1090 Steel
Current values are proportional to area

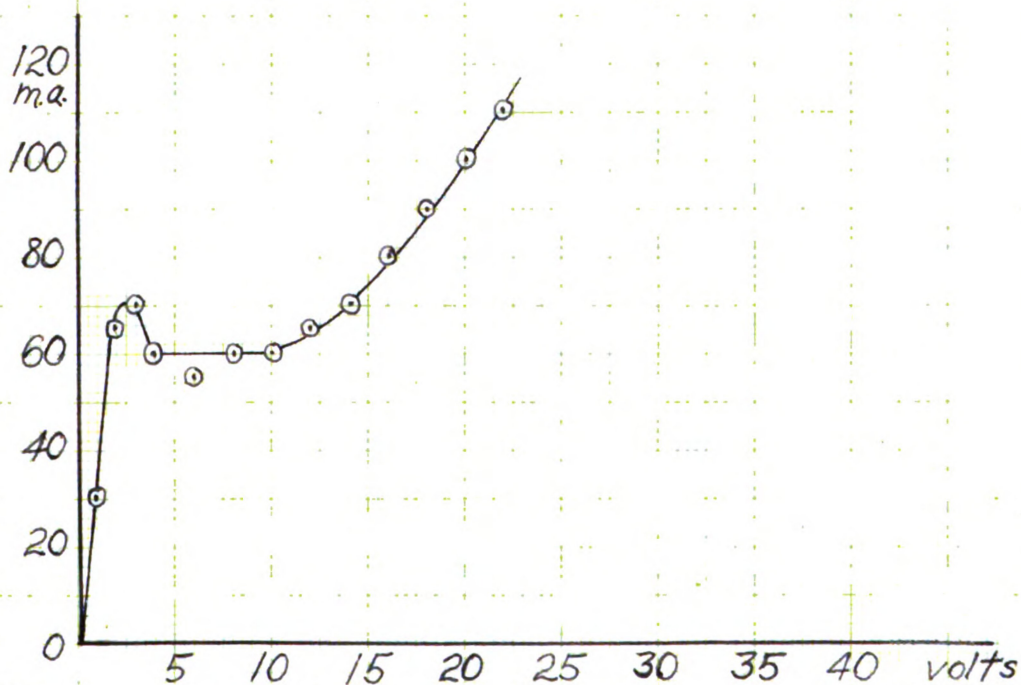


Figure 37. Voltage-Current Curve of 10120 Steel

was to shut off the stirrer while a reading of the voltmeter and ammeter was taken, restoring agitation between readings to approximate conditions maintained while polishing. Plotting voltage against current, the curves for the steels studied are drawn in Figures 34-37. For two determinations, different areas of contact will manifest themselves in two current values for the same voltage, but the breaks in the curve in both cases will occur at the same voltage. Since these breaks are what determine the range of polishing, this method is successful for securing the information desired.

The curves so secured show the typical flat portion where, according to theory, successful electrolytic polishing occurs. To test this, photomicrographs were made of SAE 1040, 1090, 1010, and 10120 steels electropolished at the voltages indicated by the curves. These photomicrographs are those of Figures 38-41.

From these illustrations it can be seen that scratches from grinding are removed and a flat even surface secured. The black spots appearing in all of these photomicrographs are not pits as would appear at first observation but are inclusions which appear above the surface. This can be shown in two ways under the microscope--by the use of polarized light and by carefully measuring the height of the inclusions by the calibrated depth of focus screw of the microscope.

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Under polarized light, the inclusions appear bright against a dark background as they reflect light which is unpolarized and therefore which passes the crossed Nicol prisms, while the light is reflected from the metal matrix still polarized. This is complicated in the examination of hardened specimens since the martensite shows anisotropy and the background metal is no longer entirely black, but enough difference in intensity remains to distinguish the inclusions.

The inclusions are not necessarily of the size of the spots, in fact are probably in all cases smaller than indicated. The inclusions distort the surrounding surface in such a way that light is reflected away from the eyepiece, and both inclusion and surrounding surface appear as a black spot. This effect may occur to a greater or lesser degree, (as will be shown later) depending upon the part of the curve to which the voltage used corresponds. The time of polishing also plays a part in this effect, for the amount of metal removed is proportional to the time of polishing and the height of the inclusion depends on the metal removed. The inclusion effect appears especially bad on photomicrographs such as these of the surface alone, but for the more widely used cases where the specimen is etched to study the microstructure, the effect is masked by the etched surface until almost unnoticeable.

Figure 38
SAE 1010
hardened
electrolytically pol-
ished at 18 volts
100x

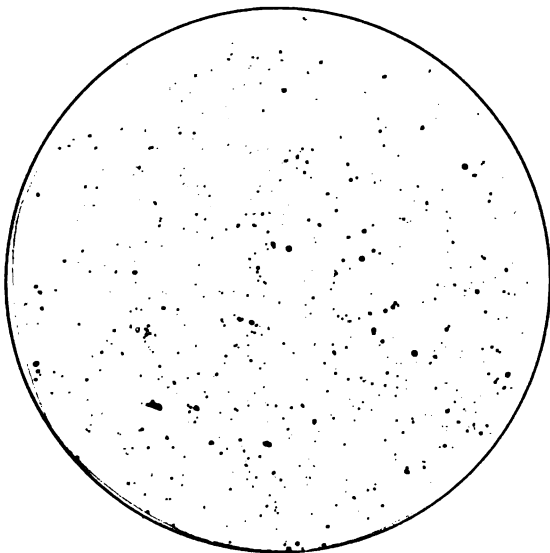
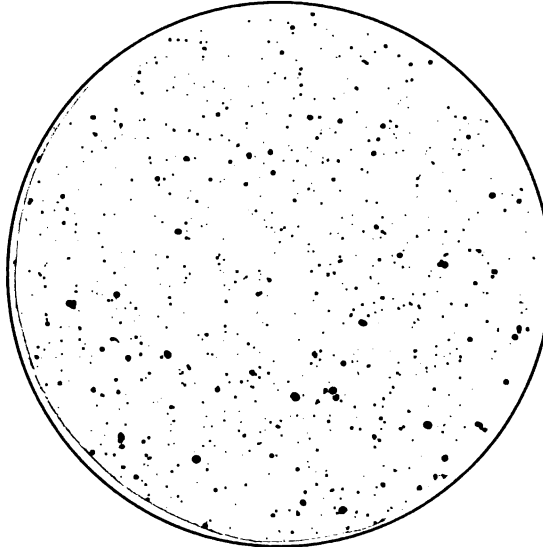


Figure 39
SAE 1040
hardened
electrolytically polished
at 16 volts
100x

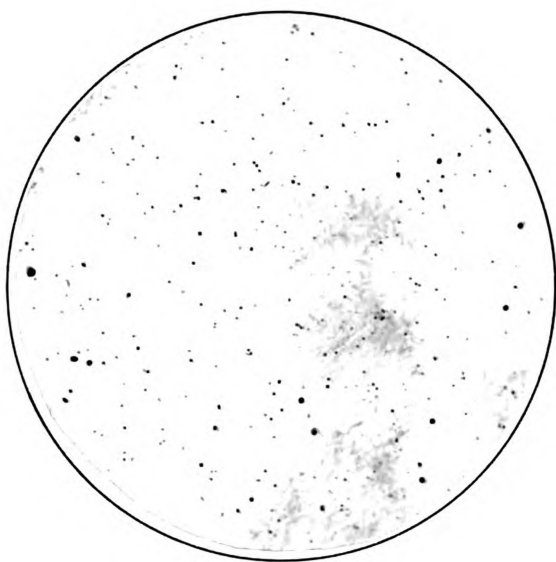
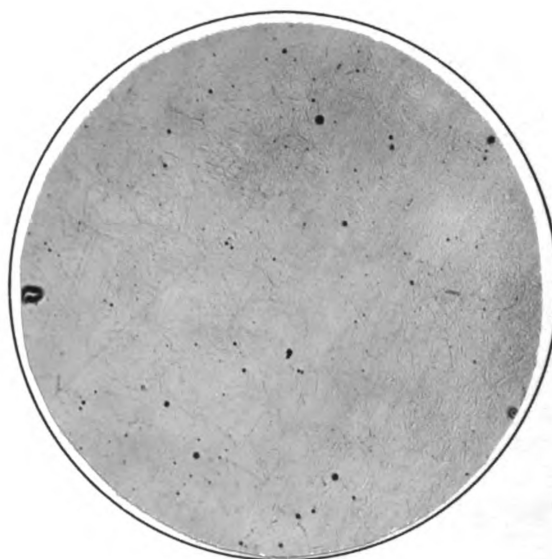


Figure 40
SAE 1090
hardened
electrolytically pol-
ished at 16 volts
100x

Figure 41
SAE 10120
hardened
electrolytically pol-
ished at 12 volts
100x



To illustrate the differences in polish obtained at voltages corresponding to different points along the curve, photomicrographs were made of SAE 1090 steel which was polished at 2,5,9,12,16,20,24,28, and 40 volts. These correspond to the points indicated in Figure 36 as listed below.

<u>Figure</u>	<u>Point</u>	<u>Voltage</u>
42	A	2
43	B	5
44	C	9
45	D	12
46	E	16
47	F	20
48	G	24
49	H	28
	I	40

Figures 42 and 43 show the result of polishing at voltages corresponding to points below the flat portion of the voltage-current curve where a viscous film is not maintained and uneven attack results. Figures 44 and 45 show the inclusion effect to a marked degree although the surface is flat and even except for the inclusions. Upon close examination, the actual inclusion may be seen in the center of the black area in many cases. Figure 50 is a photomicrograph at 750 diameters of one of the inclusions and the surrounding distorted surface of Figure 45. The presence of many very small inclusions is shown in this photograph. This shows one of the disadvantages of polishing electrolytically

as opposed to mechanical polishing, for in the latter inclusions are removed with the metal instead of being left on the surface.

Figures 40, 46, and 47 illustrate the degree of polish obtainable at the upper portion of the current-voltage curve and in the higher values of the flat portion. Here the inclusion effect is minimized and a good polish obtained.

At higher voltages, surfaces as shown in Figures 48 and 49 were obtained. An etching effect is beginning to show while at 40 volts, the inclusions are prominent, probably because of the larger amount of metal removed. Under polarized light, metals polished at high voltage values show the anisotropy of martensite without further etching better than polishes at low voltages. According to Jacquet⁵, at voltages corresponding to points above the flat portion of the current-voltage curve, gas is evolved which causes uneven attack. As no gas bubbles are observable in this electrolyte at any voltage, it is presumed that this complication does not occur in this case and polishing may proceed at higher voltages.

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Figure 42
SAE 1090
hardened
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100x

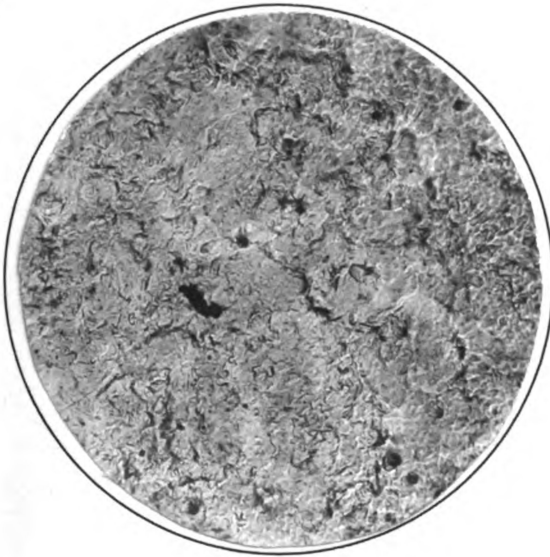
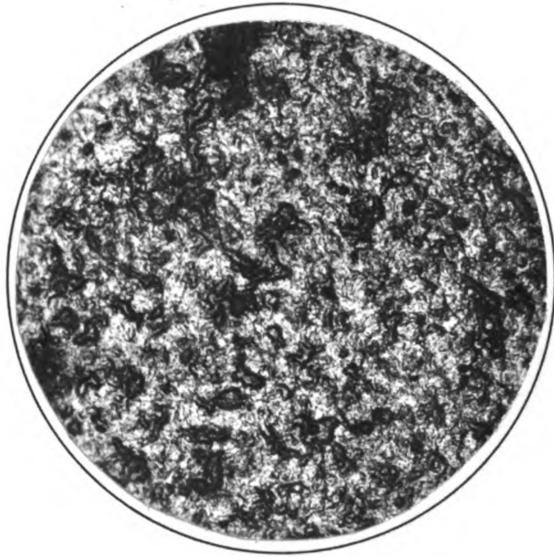


Figure 43
SAE 1090
hardened
electrolytically pol-
ished at 5 volts
100x

Figure 44
SAE 1090
hardened
electrolytically pol-
ished at 9 volts
100x

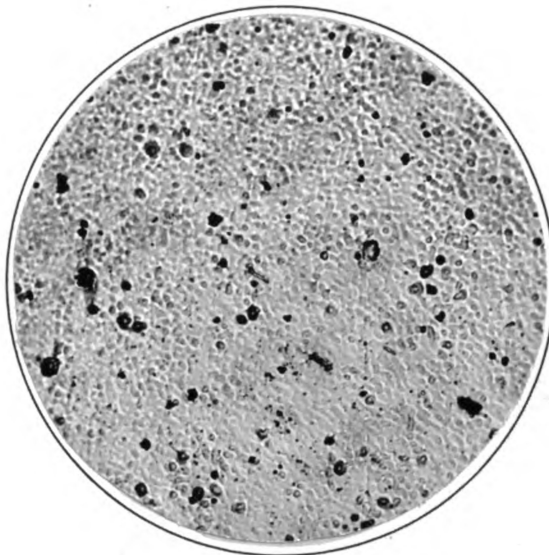


Figure 45
SAE 1090
hardened
electrolytically pol-
ished at 12 volts
100x

Figure 46
SAE 1090
hardened
electrolytically pol-
ished at 20 volts
100x

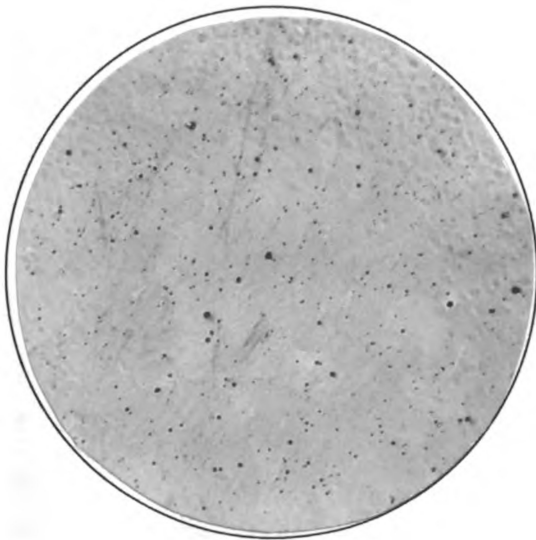
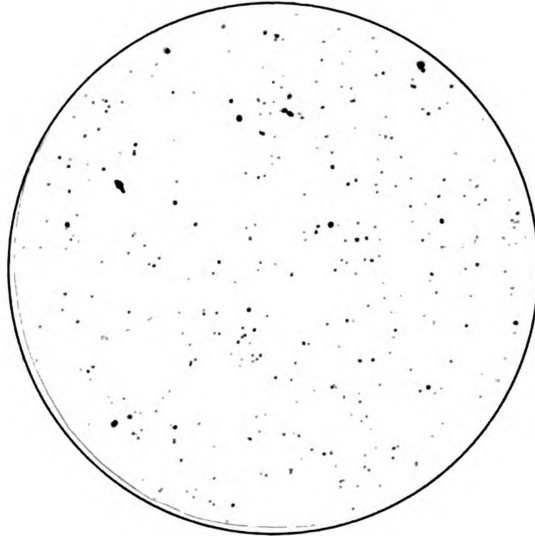


Figure 47
SAE 1090
hardened
electrolytically pol-
ished at 24 volts
100x



Figure 48
SAE 1090
hardened
electrolytically pol-
ished at 28 volts
100x

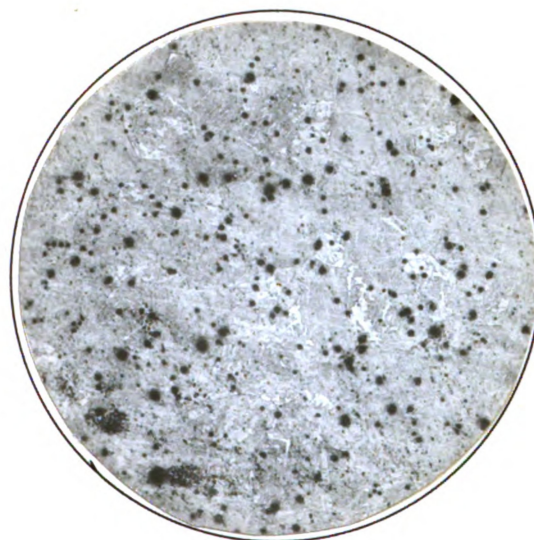


Figure 49
SAE 1090
hardened
electrolytically pol-
ished at 40 volts
100x

(50)

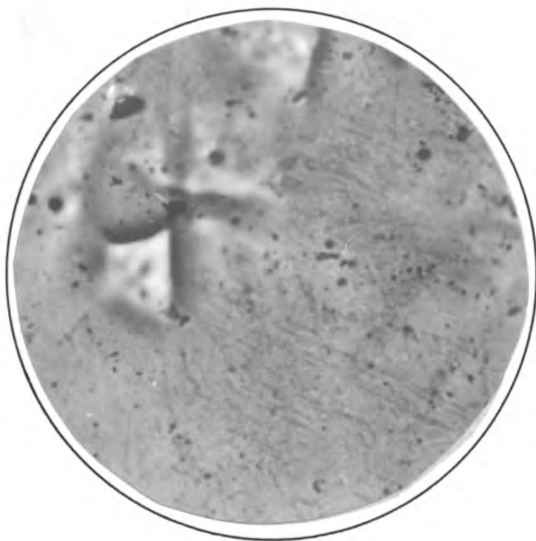
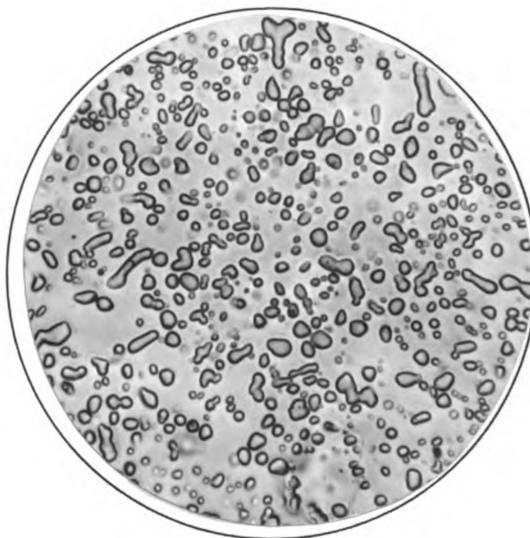


Figure 50
SAE 1090
hardened
electrolytically pol-
ished at 12 volts
750x

Figure 51
SAE 10120
spheroidized
electrolytically pol-
ished at 12 volts
750x



The composition of a steel is not the only factor affecting the voltage range, for the phases present affect the voltage values to be used and the quality of the resulting polish. Figure 51 is a photomicrograph of the same SAE 10120 steel as shown in Figure 41, but in the former the cementite is in a spheroidized condition while the latter shows a fully hardened structure. Both were polished at approximately the same voltage, but this is often not the case. The etching effect of Figure 51 is caused by the different rate of attack on the two phases, leaving the carbide in relief. The following table gives the voltage ranges for two steels with two different structures for each.

SAE 1090 annealed	2-10 volts
1090 hardened	6-20
SAE 1040 annealed	8-22 volts
1040 hardened	10-26

While the differences in range may be small as in the second case, they may also be of considerable significance as in the first.

The degree of polish obtained depends to a great extent upon the freshness of the acetic anhydride-perchloric acid electrolyte, for as it ages, the surface obtained is more or less strongly etched. This deterioration may be caused by oxidation of the organic compounds, absorption of water, or both. The solution

darkens with age which supports the first explanation, while the addition of water was observed to decrease the quality of the polish obtained thereafter.

The next series of pictures were made from specimens of SAE 1040 steel which were polished in an electrolyte which had been used for some time and resulted in etched surfaces. The specimens were different structures and hence vary considerably in the surface revealed. In addition, the same surfaces were lightly etched with 2% nital to show the variation of the electrolytically etched to the fully etched surfaces. The decrease in prominence of surface irregularities is also, upon etching, brought out in these photomicrographs of the surfaces after etching with nital. Figures 52 and 53 are photomicrographs of hardened structure, Figures 54 and 55 of annealed structure, and 56 and 57 of normalized structure. The "wavy" appearance of ferrite grains in the annealed and normalized structures and the etched needles of the hardened structure are apparently indications that the electrolyte has deteriorated and its usefulness impaired. Staining of the surface when the specimen is withdrawn from the electrolyte also is noticeable when the latter has deteriorated but does not result when using fresh electrolyte.

Figures 58 and 59 are photomicrographs at 100 and 375 diameters of SAE 1090 steel which was quenched at a rate less than the critical rate for full hardening

so that a structure consisting of both martensite and troostite resulted. The polish was accomplished in a fresh solution of electrolyte and shows the etching of the troostite while the martensite was not etched by the electrolytic polishing. The rate of attack is the same for both phases as neither is in relief and a smooth but sharply contrasting surface is obtained.

Figure 52
SAE 1040
hardened
electrolytically pol-
ished at 26 volts
375x

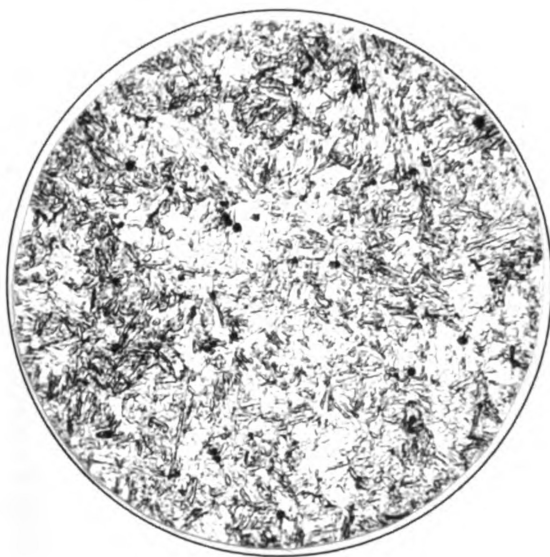
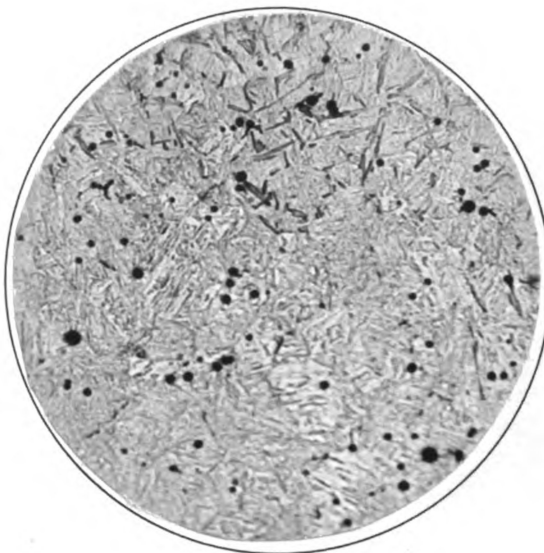


Figure 53
SAE 1040
hardened
electrolytically pol-
ished at 26 volts
nital etch
375x

Figure 54
SAE 1040
annealed
electrolytically pol-
ished at 20 volts
375x

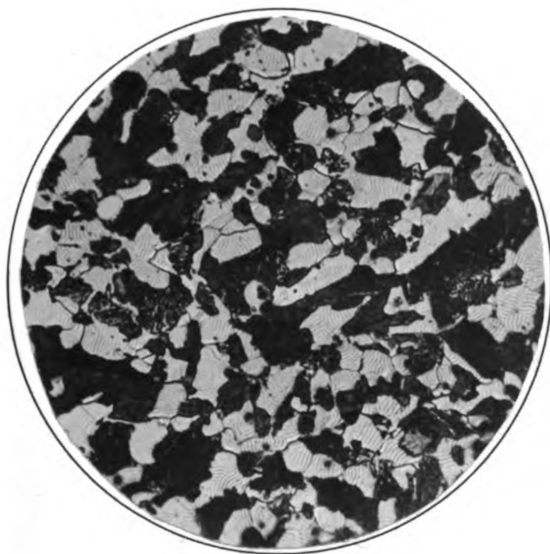
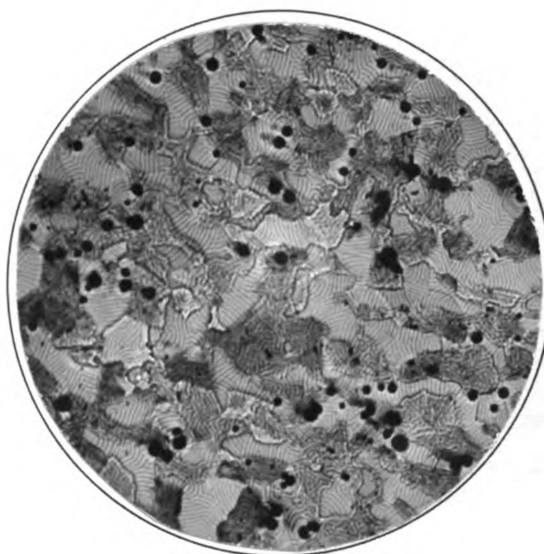


Figure 55
SAE 1040
annealed
electrolytically pol-
ished at 20 volts
nital etch
375x

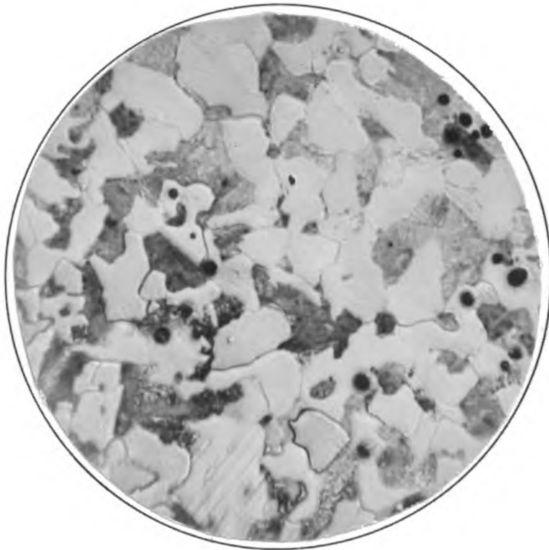
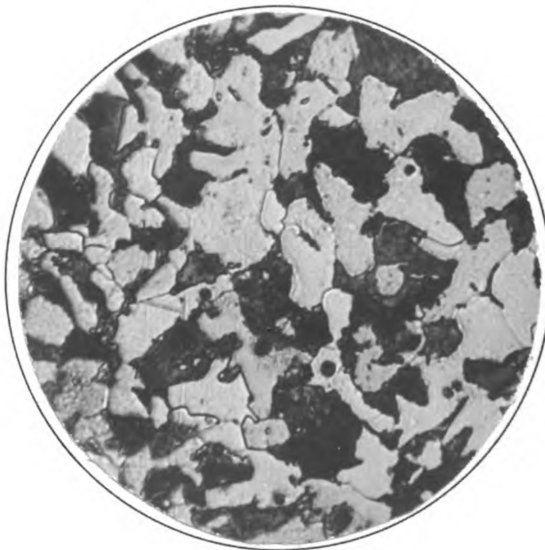


Figure 56
SAE 1040
normalized
electrolytically pol-
ished at 18 volts
750x

Figure 57
SAE 1040
normalized
electrolytically pol-
ished at 18 volts
nital etch
750x



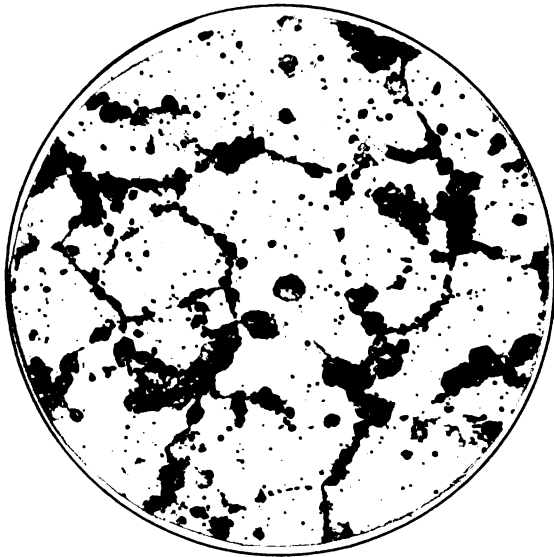
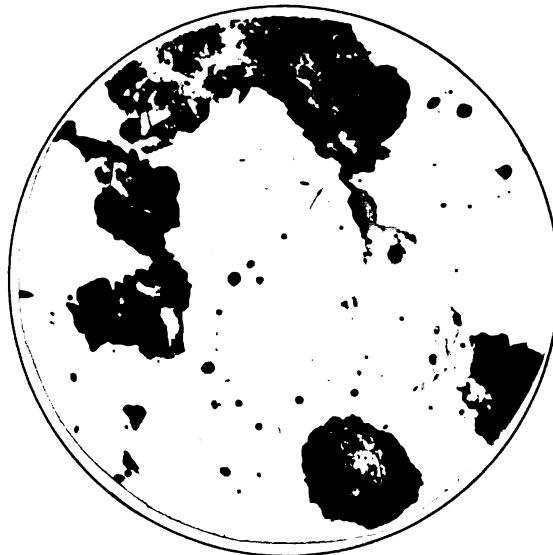


Figure 58
SAE 1090
slow quenched
electrolytically pol-
ished at 24 volts
100x

Figure 59
SAE 1090
slow quenched
electrolytically pol-
ished at 24 volts
nital etch
350x



Summary and Conclusions

The variables investigated and the results obtained or observations made will be discussed individually for each variable.

Electrolyte- The acetic anhydride-perchloric acid solution, which was the only one used extensively, was found to be successful when used fresh, but after several weeks its effectiveness diminishes. Then it is no longer possible to secure a smooth surface for etching and staining occur.

Current Density- Because of the setup of the laboratory apparatus used, the current density was not controlled. It was found to decrease when the voltage was held constant after the polishing had proceeded for a few seconds, indicating the formation of a film which retards the passage of current.

Voltage- The following table gives the kinds of steel polished electrolytically, the range of voltages for successful polishing according to current-voltage curves, and the voltages which were successfully employed in obtaining a good polish.

<u>Steel</u>	<u>Voltage Range</u>	<u>Voltage Used</u>
SAE 1010	16-30 volts	20 volts
SAE 1040 annealed	8-22 volts	16 volts
1040 hardened	10-26	16
SAE 1090 annealed	2-10 volts	
1090 hardened	6-20	16 volts
SAE 10120 normalized	2-12 volts	12 volts
10120 hardened	2-12	12

UNITED STATES DEPARTMENT OF AGRICULTURE

Report of the Secretary of the Department of Agriculture
to the President of the United States
for the year 1900

The Secretary of the Department of Agriculture
has the honor to acknowledge the receipt of
the report of the Secretary of the Department of
Agriculture for the year 1900, and to
transmit herewith to the President of the
United States a copy of the same. The report
contains a full and complete statement of
the work of the Department during the year
1900, and of the progress of the various
branches of the Department. It also contains
a statement of the financial condition of the
Department, and of the results of the
various investigations conducted during the
year. The report is a valuable document,
and it is hoped that it will be of great
service to the President and to the
people of the United States.

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of the financial condition of the Department,
and of the results of the various
investigations conducted during the year.

In general, it was found that the method of controlling the polish by means of the voltage applied was quite successful. Determination of the voltage to be used by means of the current-voltage curves was verified, as a satisfactory polish was secured in the ranges indicated in all cases. Because of the inclusion effect, it was observed that the higher values indicated minimized this effect and gave a better surface.

Time- The time factor was not investigated extensively, for it is dependent upon the current density which in turn depends upon the voltage. Enough time must be allowed to remove sufficient metal to secure a smooth surface below any scratches present, so that the time is also dependent upon the depth of the scratches. As the specimens were all polished from 4/0 emery paper, the depth of scratches was the same in each case. Five minutes was found to be long enough in most cases, but where a low current density exists and the surface is large, this time may be doubled. Over polishing results in an increase in the inclusion effect, as more inclusions are left in relief, and in increased etching of the metal itself. The optimum time for polishing would be just enough to remove all scratches. For a given metal, electrolyte, and degree of previous polish, it should be possible to construct a table from experiments that would enable one to choose the proper length of time

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according to the current density existing and the area of the surface.

Agitation- The agitation throughout these experiments was provided by a motor-driven glass stirrer of the propellor type. A stirring rate of about 150 r.p.m. was used in all cases. Stirring was found to be necessary to avoid the appearance of prominent ridges or undulations instead of a flat surface.

Temperature- While no definite data was collected on the effect of temperature, the best polishes were obtained when the electrolyte was cooled by an external ice bath to below 10°C. The electrolyte was observed to resist deterioration better when polishing was done in an ice bath instead of at room temperature. The effect of temperature may be attributed to better film maintenance because of a lower rate of diffusion at low temperatures. This would be especially so during polishing as considerable heat is evolved by the passage of the current which could conceivably have a strong influence on the film if the electrolyte were not cooled.

Cathode- The cathode used throughout the experiments was of stainless steel and about two inches in diameter. Its position was held constant: parallel to the surface being polished and at a distance of one-half to one inch.

Conclusions- While electrolytic polishing is not a fool-

proof substitute for mechanical polishing, it does offer a rapid method of securing a scratchless, strain-free surface after more or less extensive preliminary experiments are made to determine the correct operating conditions. After this is done, the saving in time of polishing and the quality of surface obtained make the method most useful.

Suggestions for Further Investigation

The most troublesome problem of electrolytic polishing is the inclusion effect, which in commercial steels containing considerable impurities greatly lowers the quality of surface obtained. It may be possible by more extensive investigation with this object in mind to greatly lessen this effect. The time factor is especially important, for the optimum length of polishing should result in a minimum of inclusions left in relief. Together with the use of voltages indicated in this thesis to be most beneficial in reducing the inclusion effect, the latter may be lessened to such a degree as to be unimportant.

Other electrolytes may be investigated as a substitute for the acetic anhydride-perchloric acid solution which deteriorates with use and must be frequently replaced. From the literature, mixtures of sulfuric and citric acids, phosphoric acid and glycerine, and methanol and nitric acid have been reported as successful and may make satisfactory substitutes.

A suggested improvement in the design of the electro-polishing apparatus is a better means for rapidly removing the specimen from the electrolyte and washing the latter from its surface in order to avoid staining.

• *Staphylococcus aureus* (Staph aureus)

• *Staphylococcus epidermidis* (Staph epidermidis)

• *Staphylococcus saprophyticus* (Staph saprophyticus)

• *Staphylococcus carnosus* (Staph carnosus)

• *Staphylococcus sciuri* (Staph sciuri)

• *Staphylococcus hyicus* (Staph hyicus)

• *Staphylococcus pasteuri* (Staph pasteuri)

• *Staphylococcus saprophilus* (Staph saprophilus)

• *Staphylococcus aureus* (Staph aureus)

• *Staphylococcus epidermidis* (Staph epidermidis)

• *Staphylococcus saprophyticus* (Staph saprophyticus)

• *Staphylococcus carnosus* (Staph carnosus)

• *Staphylococcus sciuri* (Staph sciuri)

• *Staphylococcus hyicus* (Staph hyicus)

• *Staphylococcus pasteuri* (Staph pasteuri)

• *Staphylococcus saprophilus* (Staph saprophilus)

• *Staphylococcus aureus* (Staph aureus)

• *Staphylococcus epidermidis* (Staph epidermidis)

• *Staphylococcus saprophyticus* (Staph saprophyticus)

• *Staphylococcus carnosus* (Staph carnosus)

• *Staphylococcus sciuri* (Staph sciuri)

• *Staphylococcus hyicus* (Staph hyicus)

• *Staphylococcus pasteuri* (Staph pasteuri)

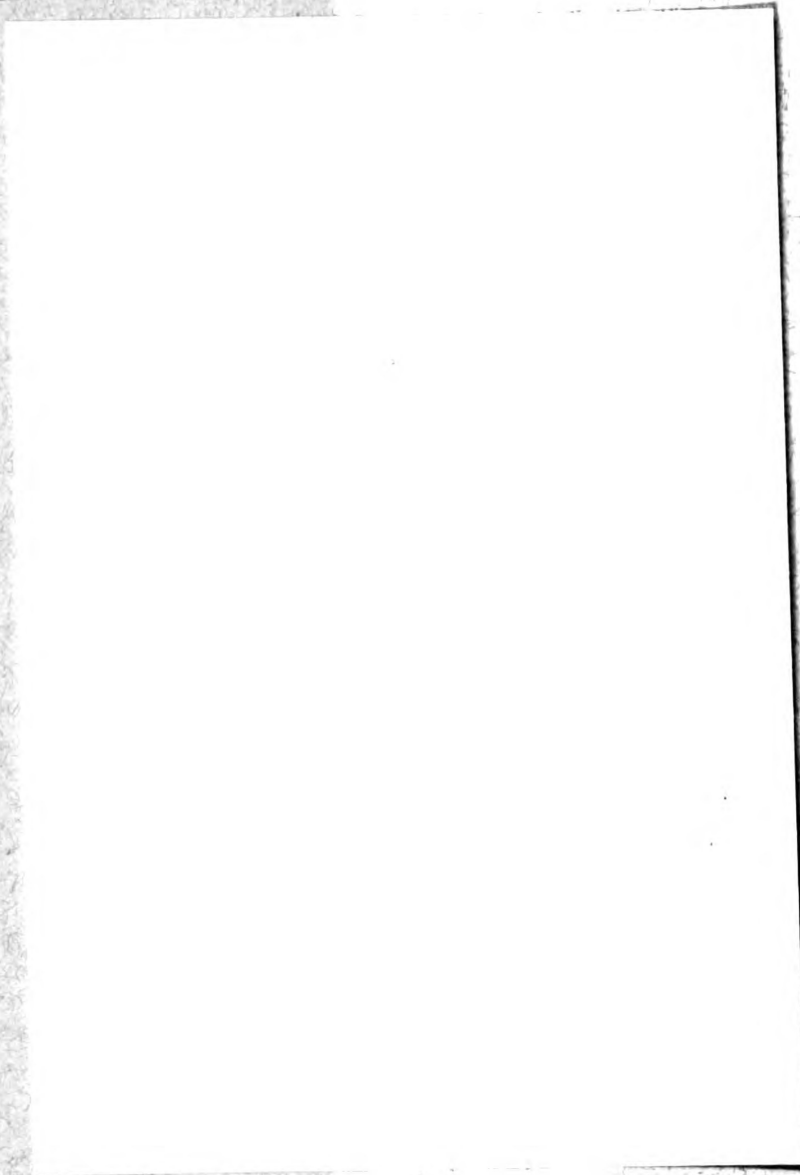
• *Staphylococcus saprophilus* (Staph saprophilus)

The extension of this type of investigation to other metals opens up an almost limitless field of work, for with the differences in compositions of the alloys and multiplicity of electrolytes possible as well as the other variable factors of electrolytic polishing, a host of experiments must be performed before the optimum conditions for each metal are determined. It is hoped that the system of investigation outlined and followed in this thesis for steel will simplify this type of investigation.

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