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REPLICATION FOR FIELD METALLOGRAPHIC EXAMINATION

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Approved by R.S. Hollingshead:
Head/Dockyard Laboratory (Atlantic) Section

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ABSTRACT

Non-destructive "replication" techniques for in-situ examination of microstructure were examined by reviewing relevant literature and theory and by doing experiments. These replication techniques involve making a replica of the surface relief of the polished and etched surface of a component with rubber or plastic. Experiments with two commercial replication media revealed that a wide range of steel microstructures could be successfully replicated. In each case, most of the information available from routine conventional (destructive) metallographic examination of the material could be extracted from the replicas.

RÉSUMÉ

Les techniques non destructrices de réplique pour l'examen in situ de microstructures ont fait l'objet d'une étude qui a porté sur la documentation appropriée et la théorie et qui comportait la réalisation d'expériences. Ces techniques de réplique comprennent une reproduction du relief de la surface polie et gravée d'un composant avec du caoutchouc ou du plastique. Les expériences effectuées sur deux répliques commerciales ont révélé qu'on pouvait reproduire avec succès une vaste gamme de microstructures d'acier. Dans chaque cas, la plupart des renseignements disponibles par l'examen métallographique classique (destructeur) du matériau pouvaient être obtenus à partir des répliques.

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EXECUTIVE SUMMARY

Introduction: In a particular metal or metal alloy, processing determines the metal or alloy microstructure and the microstructure determines the metal or alloy properties. By examining the microstructure of a component, a metallurgist can comment on the processing history of a component, its properties and its suitability for continued use. Normally examination of the microstructure of components is done destructively. A metallography technique known as replication allows characterization of microstructures non-destructively. This technique can be used outside of the laboratory, for example onboard ships, to obtain information about material structure in situ. In this study literature on replication for optical microscopy and relevant theory was reviewed; the most promising procedures and equipment for use by the Canadian Navy were identified; and experiments to determine the capabilities and the limitations of the two most suitable replicating media, one based on aluminum backed cellulose acetate films and the other based on a two component rubber media, were carried out.

Principal Results: Experiments on AISI 1080 and 4340, ASTM A210 and A336 grade F22 and HY-80 and HY-100 steels showed that much of the information available from conventional destructive methods could be extracted from the replicas. Pearlite, ferrite plus pearlite, martensite and martensite plus ferrite structures were examined. The martensite and dual phase martensite plus pearlite structures were most difficult to replicate. Even for these difficult microstructures, the phases present could be identified, their volume fraction can be estimated and the grain size can be determined. For most purposes, this is adequate, particularly when combined with knowledge of properties and composition, from in situ and nondestructive hardness testers and chemical analysis units. In general, the technique should be applicable to any material where contrast in optical microscopy can be produced by a mechanism which gives surface relief. Similar information was available from both types of replica investigated in this work. The rubber replicas proved easier to use while cellulose acetate replicas gave better looking micrographs.

Significance of Results: The ability to nondestructively assess microstructure in situ has been used by the electric power generation industries and other navies to assess the fitness of components for service. It has also been used as part of the basis for justifying the life

extension of old structures. The technology is useful for assessing the state of components which may have been incorrectly heat treated or damaged by fire or incorrect use. In warranty disputes and failure analysis the technique allows evidence to be collected before a component is returned to the manufacturer or repaired, for example, by welding. Replication has been used in investigations of cracking on the propeller blades of the Canadian Patrol Frigates and of inadequately heat treated hull gland castings on the Oberon Submarines.

Future Work: This technique is now being used, when required, in investigations of Canadian Navy problems. No further research is planned, though DREA is continuing to investigate related techniques to learn about the nature of materials in situ.

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1.0 INTRODUCTION

Engineers charged with maintaining defence equipment such as ships and weapon systems must know if the microstructure of metallic components has been altered. Such microstructural alteration, which causes changes in mechanical and other properties, may occur as a result of long term use at elevated temperature, fire damage, or incorrect installation or repair procedures. Traditionally the evaluation of the microstructure of suspect components involved destructive sectioning followed by microscopic examination. At best, this is an expensive process which requires repairs. At worst, because an appropriate repair procedure does not exist, the component cannot be inspected. In this latter case, the suspect component must either continue to be used despite a risk or else must be replaced.

Over the past decade, a field metallographic technique known as replication has been used successfully by several industries to overcome the problems just mentioned. This technique does not require sectioning of the component. Instead, as sketched in Figure 1, the technique involves grinding and polishing a sample of interest to a mirror finish, etching the polished surface to produce microstructural contrast, replicating this surface and then examining the replica of the surface with a microscope. The initial rough grinding of the surface to be examined is required to remove paint, corrosion products, and/or decarburized layers, so that the region examined is representative of the bulk. It also removes gross surface deformation or other damage. Further grinding with successively finer grit papers is done to remove the deep scratches and deformed material produced during the coarse grinding process. Polishing, either by mechanical or electrochemical means, is then done to remove the layer of cold worked material produced by the grinding process and to produce a smooth surface suitable for etching. Following polishing the surface is etched with an etchant which reveals the microstructural features by producing surface relief. A metallographic replica of the etched microstructural features is then made by either pressing a solvent softened replica foil or film onto the surface or by placing liquid rubber onto the surface. When hardened, the replica is removed and mounted on a microscope slide. This replica retains all of the relief of the surface as a negative image and when examined with an optical microscope provides much of the information obtainable by conventional optical metallography, without the associated specimen destruction [Simmons, 1987; Neri,1969].

Replication, though conceptually straight forward and successful in a number of related applications, has been little used by the Department of National Defence. This is a result of a lack of experience and a consequent lack of confidence in the technique, a lack of sufficient data on the replication of materials of interest, and a need to be able to adapt the technique rapidly to materials not previously investigated. The work described herein was undertaken to overcome these problems.

To achieve this goal, literature on replication was reviewed in order to assess the technology and the details of the techniques used by others. Together with the relevant scientific background, this provided a base upon which to clarify requirements and to

develop techniques with which to satisfy them. The techniques thus developed were evaluated by comparing the results of conventional and replica metallography. Analysis of this compendium of conventional and replica micrographs allowed the capabilities and limitations of the technique to be evaluated.

2.0 LITERATURE REVIEW

The technique of replication, which was first suggested by Haycraft[1] [1891], is now used to examine metal surfaces in transmission electron microscopy [Goodhew, 1985], scanning electron microscopy [Gabriel, 1985] and optical microscopy [Henry et al., 1987]. Field metallography may involve the use of all of these replica techniques, but for many applications, optical microscopy is sufficient. Preparing replicas for examination in the optical microscope and examining is thus the focus of this report. The method of producing optical replicas was summarized earlier. In the remainder of this section, the microstructural information which can be obtained by replication and the applications of replication are considered.

Replication has been used to determine the phases present in a material and their relative amounts [Hedgecock et al., 1985], to examine microstructural features such as grain size, grain boundaries, carbide particles, graphite sizes and distribution, and pearlitic lamellae [Nee, 1989] and to record high temperature phenomena such as creep cavitation, spheroidization and precipitation of alloying elements from solid solution [Sullivan, 1987]. As well, replication has been used to identify the location [Thielsch, 1991], nature (e.g. fatigue, stress corrosion, intergranular) [Marder, 1985], and origin of cracking [Brown and Smith, 1982]. It has also been used to follow the progression of a microstructural feature over time (e.g. crack propagation, creep cavity formation) [Gabriel, 1985]. The technique can also be used in hostile environments. For example, cellulose acetate replicas have been taken from components at temperatures as high as 150°C [Birner and Lohberg, 1980] and rubber media replicas can be used to perform underwater metallurgical inspections [Nee, 1989]. Thus replication can be used to characterize a range of metallurgical microstructures under a wide range of conditions [Gleiter, 1983].

Microstructural assessment by replication is used to support failure analysis investigations [Thielsch, 1991] and to provide information to assess the remaining life and/or to suggest repairs appropriate for extending the life of components used beyond their design life [Henry et al., 1987]. It has been used in a range of engineering applications on such materials as carbon, alloy and stainless steels, cast irons, various aluminum alloys, Ti-6Al-4V, and brasses and bronzes [Nee, 1989; Brown and Smith, 1982; Simmons,1987 and Hedgecock et al., 1985]. Some well documented examples of successful uses of the technology are listed in Table 1. The first two—creep damage assessment and crack evaluation—warrant further discussion.

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[1] Dr. Haycraft took excellent micrographs of collodion replicas of muscle tissue at magnifications of up to 1000 times. A noteworthy result for his time.

Table 1. Some Applications of Replication

Application	Reference
1. Creep cavity detection for the assessment of damage and the evaluation of remaining life of high temperature components.	[Viswanathan, 1985,1989] [Balaschak and Strauss, 1987] [Henry et al., 1987] [Ludwigsen, 1987] [Thielsch, 1991]
2. Metallographic investigation of cracking to determine the cause and nature of the cracking.	[Cervoni and Clark, 1987] [Ludwigsen, 1987] [Thornly and Sedman, 1990] [Marder, 1985]
3. Metallurgical assessing to determine component fitness for service in the aerospace industry of aluminum and titanium alloys.	[Neri, 1969]
4. Assessment of super alloy turbine blades.	[Wood, 1990]
5. Identification of fatigue crack initiation site in a Ti-6Al-4V alloy.	[Brown and Smith, 1982]
6. Study of crack micro-geometry in slow-propagating fractures.	[Conor, 1972]
7. The detection of precipitates in high temperature components.	[Marder, 1985]
8. Measurement of surface roughness.	[Smolvschowski, 1946]
9. Determination of microstructural alteration following fire damage.	[van Sevenhoven, 1990] [Weldler and Neubauer 1979]
10. Microstructural assessment of the quality of a weld.	[Simmons, 1987]
11. Examination of wear debris in ship engines.	[Eyrie, 1981]
12. Weldment failure analysis.	[Vrengde, 1988]
13. Differentiation between cast iron and cast steel.	[Nee, 1989]
14. Determination of heat treatment of ferrous alloys.	[Simmons, 1987]
15. Detection of matrix cracks in composite laminates.	[Masters, 1987]
16. Replication and identification of cracks with magnetic rubber.	[Weltman et al., 1989]

The most widespread and significant use of replication has been to assess the creep damage state of high temperature components—boilers, boiler tubing, steam piping and headers etc.—made from low alloy steels [Viswanathan, 1989; Henry, 1987; Wood, 1990]. Coupled with an extensive data base for the alloys of interest and appropriate engineering decision making, this provides the basis for a monitoring and maintenance program which allows the utilization of the full life of fossil fuel power generating plants [Viswanathan,1989]. Since this may be significantly longer than the design life, cost savings are realized as a result of such creep damage assessments. With replication, creep damage is assessed by noting that certain regions, for example, coarse grained heat

affected zones of welds, regions of high stress and regions known to have failed previously, are most susceptible to creep cavitation. Replicas taken from these regions are then examined to determine if creep damage has accumulated and if it has, to assess its severity. As shown in Figure 2, the very early stages of creep damage require only periodic monitoring, while the latter stages require immediate replacement. Regardless of what is learned from assessments by replication, it provides a better picture of the state of components than design life estimates which are often inaccurate because of lack of material data and uncertainty about operating temperatures.

In addition to evaluating the severity of a known type of cracking in a known location, as in the creep damage application just described, replication can also be used to determine the origin of cracks detected by traditional non-destructive testing techniques such as magnetic or florescent particle methods. As shown in Figure 3, different types of cracks have different propagation patterns and these propagation patterns can be recorded on a replica of a polished surface. Once recorded, these propagation patterns can be used to identify the crack type. Since the type of repair required, as well as the changes required in the way the repaired component is to be used, depend on the type of cracking observed, this information is useful in engineering decision making [Ludwigsen, 1987]. For example, by using replication, Thornly and Sedman [1990] determined that cracking detected during non-destructive examination of a hydro-turbine had not newly developed but was hot tearing that had occurred during manufacturing. As the component had been in operation for 40 years, the cracks were not regarded as detrimental and expensive replacement costs were avoided. Similar applications include characterization of cracking in aerospace components [Neri, 1969], turbines in nuclear generating stations [Cervoni and Clark, 1987], warship propellers [Hedgecock et al., 1985] and in generator retaining rings used at electrical generating stations [Thornly and Sedman, 1990].

While replication has been successfully used in the variety of applications and on the variety of materials mentioned above, some problems in applications envisioned important to DND have yet to be solved. Most important is the lack of work on ferrous alloy microstructures such as martensite and dual phase martensite plus ferrite. It is unclear if this is just because these microstructures are rarely observed in the alloys studied to date or because there are experimental difficulties in dealing with these microstructures.

3.0 SCIENTIFIC BACKGROUND ON PROCEDURES

To address the problem just mentioned, to evaluate the variety of techniques published in the literature and to develop procedures appropriate for the applications envisioned for DND, it is necessary to consider in more detail the key procedures: polishing, etching and replication.

3.1 POLISHING

Polishing may be done by either electropolishing or mechanical polishing [Sullivan, 1987]. Electropolishing has two advantages over mechanical polishing. It is operator insensitive

and produces a final surface free of metal deformed by the cold work associated with mechanical polishing. This latter feature is most important for soft metals which are most susceptible to this type of damage. The principal disadvantage of electropolishing, especially for applications which involve use in confined spaces, is safety. In at least one case, a portable electropolishing unit filled with perchloric acid and methanol caused a fire during use. Thus such units are not suitable for use in confined spaces. Even in the demanding application of creep damage assessment, where electropolishing produces superior results according to some workers, mechanical polishing can be acceptably substituted. It is in this light that procedures specifying electropolishing should be viewed. Except in the case of very soft metals, the substitution of mechanical polishing should be possible, making the process acceptable for the confined spaces found, for example, on board a ship.

3.2 ETCHING THEORY

Etching is the step in field metallographic replication which determines, more than any other, what information will be present in the replica. In contrast with conventional metallography, where etching can be used to produce contrast by a variety of mechanisms, the only contrast produced by etching which can be replicated is surface relief. Surface relief can be produced by mechanisms such as grain contrast etching, grain boundary etching and duplex electrochemical etching [Kehl, 1949]. This occurs in both chemical and electrolytic etching, though not in all etchants of these types [Gleiter, 1983]. As well, ion-bombardment etching may produce sufficient surface relief to be included in this category [Vander Voort, 1984]. To identify which etchants are appropriate for replication, to optimize the replication process and to understand the results of this work, one must consider how relief etching occurs in each of the relief etching mechanisms mentioned above.

3.2.1 GRAIN CONTRAST ETCHING

Grain contrast etching is one of the two mechanisms by which relief etching of single phase materials takes place. It involves dissolution of the specimen by the etching solution by a stepwise process (analogous to facetted melting [Porter and Easterling, 1981]). This type of etching produces a series of differently angled facets which are the same on any one grain but which vary from grain to grain. As shown in Figure 4, the angle of the faceting determines to what extent the incident light rays will be reflected or scattered outside the microscope aperture and thus their relative brightness in the microscope. This phenomenon, which is known as oriented-grain lustre, [Kehl, 1949] can be easily replicated provided the preferential etching effect is strong enough.

3.2.2 GRAIN BOUNDARY ETCHING

The second single phase etching mechanism, grain boundary etching occurs because of grain boundary segregation. The difference between the grain boundary and bulk composition gives rise to a potential difference [Vander Voort, 1984], with the normally

more electropositive boundary being anodic to the bulk. Thus preferential dissolution of the boundary occurs. The degree of impurity segregation has a significant influence on the rate of etch response [Vander Voort, 1984]. In the absence of any segregation or other significant change in grain boundary chemical potential, little grain boundary etching occurs.

3.2.3 DUPLEX ELECTROCHEMICAL ETCHING

Duplex electrochemical etching is the dominant mode of etching in most multiphase microstructures. It is essentially a controlled corrosion process resulting from electrolytic action between two or more phases with different electrochemical potentials. The more electropositive phase behaves as the anode and is preferentially dissolved to below the original plane, and may appear darkened due to shadow effects. The more electronegative phase behaves as the cathode and remains undissolved and stands in relief [Kehl, 1949]. Because the more anodic phases will etch more quickly than the more cathodic phases, each phase present in multiphase alloys will become optimally etched after a unique etching time, different than that required for the other phases. As well because of the larger potential differences driving the process, the kinetics of duplex electrochemical etching are normally faster than those of the two single phase mechanisms just mentioned. Thus a common difficulty with multiphase alloys is to produce optimal feature development of all phases following a single etch. This difficulty is often overcome in conventional etching with tint and other non-surface relief etchants. Such etchants either stain different phases different colors, or produce an anisotropic or interference surface layer which allows contrast to be developed without relief. So far, such non-surface relief techniques have not been used with replication and it seems unlikely that they will be.

3.3. REPLICATION MATERIAL AND METHODS

A variety of replication materials and methods have been developed for optical replication. Of these, techniques based on roughly 100 μm thick cellulose acetate tape softened in acetone or methyl or ethyl acetate(Viswanathan, 1985), are most commonly reported. There are two problems with these replicating methods. First, successful replication requires careful control of pressure during replication, which in turn requires some operator experience and skill. Second, to exploit all the information in cellulose acetate replicas, which are transparent, it is necessary to vacuum coat them with a layer of reflective metal prior to examination. This step can make the replication process prohibitively long and complex [Kosec and Vodopivec, 1969]. The second problem has been eliminated by Struers and perhaps other manufacturers, who provide a replication kit based on cellulose acetate precoated on the backside with a reflective metal foil. A variety of other plastic replication materials are also available [Wilson and Rowe, 1980]. However, most are not sufficiently robust for field metallography.

In addition to the replication methods based on plastics, there are also several based on rubbers, including silicon rubbers, rubbers containing magnetic particles and dental impression media, all of which are non-transparent and thus do not require coating.

Traditionally these have been used for crack and fracture surface replication. Recently a commercial system based on a two part synthetic rubber and a simple to use application system which requires little operator skill has been developed. According to the manufacturer of this system, it is capable of replicating details with a resolution of better than 1 μm.

4.0 PROBLEM DEFINITION

The forgoing literature review suggests that replication can be used to non-destructively assess the microstructures of most steels with confidence. Cases where there is insufficient data to make this conclusion are fine grained martensite and dual phase steel microstructures. Confident use of replication techniques by DND will require:

1. Replication materials and procedures which give an acceptable combination of safety and portability in the confined quarters aboard a ship, ease of use in all positions including vertical and overhead, accuracy and repeatability, and operator and environmental insensitivity; and

2. Convincing evidence of the capabilities and limitations of replication, especially for martensitic steels.

The specific problems just mentioned were solved by adapting and developing two replication techniques. As discussed in more detail below, the techniques involve mechanical polishing, etching with 2 % nital using a proprietary device which allows all position etching with no spillage, and replication with the commercial replication media made by Struers and Microset. Capabilities and limitations were assessed by comparing the results of replication techniques to those of conventional metallography on a range of steel microstructures including a variety of martensitic microstructures.

5.0 MATERIALS AND METHODS

5.1 MATERIALS

A number of steels with a variety of microstructures were examined. They included:

1. AISI 4340 and ASTM A336 grade F22 steels with both martensitic as well as dual phase martensite plus ferrite microstructures;

2. ASTM A210 grade A-1 pressure tube steel, with a fine pearlite plus ferrite microstructure;

3. AISI 1080 steel with a pearlitic microstructure; and

4. HY 80 and HY 100 submarine pressure hull steels, with low carbon tempered martensitic microstructures.

The chemical composition of the steels used are given in Table 2.

Table 2. Steel Compositions

Element	AISI 4340	HY 80	HY 100	ASTM A210	AISI1080	ASTM A336 grade F22
	(limits)	(specimen used)	(limits)	(limits)	(limits)	(limits)
Carbon	0.38-0.43	0.17	0.20 max	0.27	0.74-0.88	0.15 max
Manganese	0.60-0.80	0.38	0.10 - 0.40	0.93 max	0.60-0.90	0.30-0.60
Silicon	0.15-0.30	0.18	0.15 - 0.35	0.10 min		0.50 max
Nickel	1.65-2.00	2.58	2.25 - 3.50			
Chromium	0.70-0.90	1.56	1.00 - 1.80			2.00-2.50
Molybdenum	0.20-0.30	0.30	0.020 -0.060			0.90-1.10

5.2 APPARATUS

The field metallographic equipment used in the experiments is shown in Figure 5. It included: for rough grinding, a standard body grinder equipped with 100 grit paper; for fine grinding and polishing, a commercial unit built for the purpose; for examination of the surface to be replicated between grinding and polishing steps and following etching, a hand size battery powered microscope; and for all position etching, a portable electropolisher-etcher. The design of the electropolisher-etcher allowed it to be positioned, used to etch a surface, and then removed with the loss of only a few ml of etchant. Replication was done on all specimens with both the Struers Transcopy Replica Kit, and the Applied Metallurgical Services Microset Replica System. The former system uses a plastic replica foil made of cellulose acetate which is softened with ethyl acetate solvent, while the latter system uses a rubber compound.

5.3 EXPERIMENTAL PROCEDURE

The experiments involved two parts. First, all the steels listed above were prepared by conventional metallographic methods and examined and photographed with a metallurgical microscope. This provided a base line against which the replicas could be evaluated. In some cases, hardness testing and tint etching, often with sodium metabisulfate, which colors ferrite white, martensite black and bainite brown, was used to characterize phases about which there was doubt. Second, replicas of the steels listed above were made.

The procedures used to prepare the steels for replication were similar for both replication methods. They involved rough grinding with a mini-grinder or a body grinder where needed, fine grinding with the handgrinder kit described above, polishing with the same handgrinder kit, and then etching to produce surface relief. To identify optimum etching

times, to understand the effects of material overetching and to evaluate incremental etching methods, an incremental etching procedure, similar to that suggested by Simmons [1987], was sometimes used. The method involved replicating the surface at a number of different etching times[2], with the hope that each phase would develop clearly at some etching time. Regardless of the exact etching procedure used, 2 % nital was generally used as the etchant, since it performed at least as well or better than the alternatives all of which are more hazardous. Following etching, and in the case of incremental etching following each etch, the surface was replicated, using either the Microset replica system or the Transcopy replica system. More detailed procedures for each of these steps are given in Appendix 1.

6.0 RESULTS AND DISCUSSION

6.1 REPLICATION OF SPECIFIC MICROSTRUCTURES

6.1.1 REPLICATION OF AISI 1080

Micrographs of 1080 Steel with a coarse Pearlitic microstructure (2 % Nital etch) are shown in Figure 6. The micrograph of the conventional specimen (Figure 6a) is very similar to that of the cellulose acetate replica (Figure 6b). Similar success was obtained with rubber replicas. In both cases replication was relatively easy to perform. The only problem came when etching times were too long. Then the cementite lamellae stood so far proud of the ferrite matrix that they broke off.

Replication of this alloy was particularly successful because:

1. etching of pearlite occurs by duplex electrochemical etching, with the cementite cathodic to the ferrite, which is preferentially dissolved by the etchant;

2. the structure is relatively coarse; and

3. it is not necessary to see details in either the ferrite or cementite phases in the pearlite to characterize the structure (in other words, only one etching process is important);

Etching multiphase structures becomes more difficult when structure in each phase must be developed by a single phase etching process while a dual phase etching process is also active and dissolving one of the phases.

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[2] Etching times are not given in this work because they are determined not only by etchant concentration and age, but also by ambient temperature and material. As with conventional metallography the user must identify appropriate etching times. A procedure for doing this is suggested in Appendix 1.

6.1.2 REPLICATION OF ASTM A210 GRADE A-1 (BOILER TUBE) STEEL

Micrographs of an ASTM A210 steel typical of that used in boiler tubes and similar equipment, are shown in Figure 7. This is ferrite-pearlite steel also replicated well with both cellulose acetate (Figure 7b) and rubber replicas (Figure 7c). The pearlite spacing is fine enough in the micrographs in Figure 7 that the pearlite laths are not resolvable. However the difference in contrast between the ferrite and pearlite can be seen in the replicas and in the conventional micrograph. This is possible because the grating like surface of the fine pearlite causes increased light scattering relative to the flatter (but also grating like) surface of the ferrite (Kehl, 1949). This relief replicated well. The slight decrease in quality of the ferrite plus pearlite replicas relative to the pearlite replica (Figure 6), is thought to be a consequence of the etching time required to produce optimum surface relief for replication dissolving enough of the ferrite away to cause it to appear at a slightly different plane of focus than the pearlite.

6.1.3 REPLICATION OF WATER QUENCHED AISI 4340

Micrographs of AISI 4340 steel water quenched from 870 °C are shown in Figure 8. The lath martensite structure is clearest in the conventional micrograph (Figure 8a). The cellulose acetate replica (Figure 8b), replicates details of the microstructures slightly better than the rubber replica (Figure 8c). More information was available by direct examination of the replicas in the microscope. Here slight adjustments in focus allowed out of focus regions, caused by the required deep etch and waviness of the replica surface, to be brought into focus. Whether directly examined or examined in micrographs, both types of replicas produced an image substantially poorer than the conventional micrograph. This is probably because an anisotropic surface film is produced on the specimen during etching with Nital[3]. Such films would contribute to contrast (by a non surface relief mechanism) on the conventional specimen, but not on the replicas. It is not clear why the rubber replica is not quite as sharp as the cellulose acetate one. Perhaps this is a result of a lower intrinsic accuracy of the rubber medium, or of contrast enhancement by interference effects causes by the reflective backing on the cellulose acetate replica.

The main questions these results raise are: for martensitic microstructure are the results obtainable from replica methods useful and, if so, what are their limitations. The micrographs of replicas shown in Figures 8b and 8c can be identified as martensitic microstructures, though many grain boundaries are missing. In previous work, quenched and tempered and air cooled specimens of AISI 4340 were replicated with similar success. In practice, these results together with knowledge of composition, could be used to determine if a structure is martensitic or not. As well, gross differences in state of temper could be identified and the grain size could be estimated. Replicas of the quality shown in Figures 8b and 8c, could also be used to identify differences in structure from one part of a component to another, though quantifying these differences would probably require work

[3] It is probably the existence of such an anisotropic surface film or films that allows polarized light to improve the contrast of martensitic microstructures in conventional metallography.

with well characterized materials. So would determining the exact state of temper, though in this regard, hardness testing could allow adequate determinations in most cases.

6.1.4 REPLICATION OF HY-80 AND HY-100 STEELS

Micrographs of HY-80 and HY-100 steels are shown in Figures 9 and 10 respectively. These quenched and tempered low carbon 'ultraservice steels' are widely used for submarine pressure hulls and for parts of warships where a very high toughness steel is required. Replication was successful on both steels. The micrographs of the cellulose acetate replicas of HY-80 (Figure 9b) and HY-100 (Figure 10b) steels are similar to the corresponding conventional micrographs (Figures 9a and 10a). The micrographs of the rubber replicas are slightly less sharp. With these steels, much longer etching times, more than double that for conventional metallography, were required to produce sufficient surface relief for replication.

The limitations of replicas made with both types of media are similar to those discussed in section 6.1.3. As well, steels such as HY-80 and HY-100 often contain both temepered martensite and bainite in their structure. However, it is unlikely that one would be able to determine relative amounts of bainite and martensite in theses material. The results of the HY-80 and HY-100 experiments, confirmed the results of earlier experiments with AISI 4340 which showed that tempered martensitic structures could also be successfully replicated.

6.1.5 REPLICATION OF DUAL PHASE MICROSTRUCTURES

For military applications, steels are seldom deliberately tempered to have a dual phase martensite plus ferrite microstructure. However, such structures might be produced during incorrect heat treatment or repair, or by overheating during use. Determining if dual phase structures could be replicated was thus an important part of this study.

Figure 11 shows micrographs of AISI 4340 steel water quenched from roughly 780°C to produce a dual phase microstructure. Figure 11a is a micrograph of a conventional specimen etched with 2 % nital. There is good delineation of the ferrite-martensite grain boundaries. However detail in the martensite regions is limited. Replicating this microstructure proved difficult. An etching time about double that for the conventional micrograph shown in Fig. 11a was required for the production of the replica micrograph shown in Fig. 11b. This replica revealed many of the ferrite-martensite grain boundaries. It might not provide enough information, in and of itself, to convince a metallographer the structure is martensite plus ferrite. The underlying question one would ask is could this instead be a ferrite plus fine pearlite structure like that shown in Figure 6. However depending on the steel and other factors, it would likely be possible to deduce this from longer etching times, coupled with in situ measurements of hardness and composition. Presuming one were convinced that the microstructure shown in Fig. 11b were ferrite and martensite it would probably be possible to estimate the volume fraction of martensite and the grain size from this replica micrograph.

Details of the martensite structure are shown in the replica micrographs in Figures 11c and 11d. Preparation of the cellulose acetate replica shown in Figure 11c required etching for four times longer than for the conventional micrograph in Figure 11a. This longer etching time caused blurring of the ferrite grain boundaries, and caused the ferrite to be out of focus when the martensite was in focus. In the micrograph and more clearly in the microscope, one can discern details of the martensite laths which allows one to conclude, from the replica, that the microstructure is probably a martensite plus ferrite one. Longer etching times before replication (Figure 11d) provided slightly more information about the martensite structure at the cost of delineation of the ferrite grain boundaries.

Acceptable results were also obtained with rubber replicas. At etching times only 20 % longer than used for conventional samples, the ferrite was well delineated in the replica as shown in Figure 12a. Longer etching times (Figure 12b) revealed detail about the martensite microstructure, while blurring the ferrite grain boundaries. While the appearance of the rubber replicas is less pleasing than that of the cellulose acetate ones, essentially the same information is available from both types of replicas.

Compared with all the microstructures mentioned above, dual phase martensite plus ferrite microstructures were harder to replicate. This is thought to be a result of etching occurring by a combination of duplex electrochemical and grain contrast etching mechanisms. According to this argument, electrochemical differences between the martensite and ferrite phases cause the ferrite to be preferentially dissolved much more quickly during etching than the martensite phase. This occurs at a rate faster than the grain contrast etching in the ferrite and especially the martensite. Thus if etching times long enough to resolve the martensite structure are used, enough of the ferrite is dissolved that one cannot, using an optical microscope, examine both phases at the same time, because of differences in the required depth of focus. This same problem makes conventional metallography difficult, though in this case the problem can be overcome by tint etching.

In summary, because two etching mechanisms are active during the etching of dual phase steels, it is difficult to produce a good replica. Replicas of dual phase structures allow the structure to be identified as probably being a dual phase martensite plus ferrite structure. However, to be certain, complimentary determinations of composition and hardness and perhaps replication of similar specimens of know composition, microstructure and thermal history may be required.

6.1.6 MICROSTRUCTURE OF ASTM A336 STEEL WITH AN UNKNOWN THERMAL HISTORY

Figure 13 shows the microstructure of ASTM A336 steel from near the neck of a large high pressure air bottle. The microstructure shown is thought to have been produced by the hot forging, quenching and tempering associated with the manufacture of the bottle followed by unintended heat treatment from repeated and incorrect welding in and cutting

out of end plugs. Figure 13a shows a micrograph of a conventional specimen etched with Nital. This appears to be a dual phase martensite plus ferrite microstructure, though in Figure 13a identification of the ferrite and martensite phases is difficult. Figure 13b shows another micrograph of a conventional specimen, this time etched with a 10 % sodium metabisufate tint etchant. This etchant colors ferrite white and martensite dark gray and allows the volume fraction martensite to be identified with certainty. Figure 13c shows a replica micrograph of this steel taken after etching with Nital. From this rubber replica one can conclude the structure is probably a martensite-ferrite dual phase structure modified by tempering. One can also discern the martensite-ferrite grain boundaries and crudely estimate the volume fraction martensite.

6.2 ARTIFACTS

Imperfections in replicas not present on the original surface are called artifacts. The most common artifact in replicas of metallic surfaces is a lack of flatness of the mounted replica over distances as long as a millimeter. This is usually a result of difficulties in mounting the replica on a supporting glass slide. This is the reason many replica micrographs in this document have a corner out of focus. Other types of artifacts, which were most common in plastic replicas, were dirt and dust trapped in the foil and bubbles in the replica film. Both are shown in Figure 14. The former can be eliminated in all but the dirtiest environments by keeping the replica foil and surface to be replicated dust free before, during and after replication. The latter can be controlled by adjusting the amount of solvent on the film and by controlling the pressure during replication.

6.3 REPLICATION SYSTEM EVALUATION

As shown in Figures 7 through 11, both replica systems produced adequate results. For the easier to replicate microstructures, such as pearlite and ferrite plus pearlite the plastic media produced more pleasing results. This was also true, to some degree, of microstructures containing martensite. In these cases, however, longer etching times were often required to produce an adequate replica with the plastic material. The reason for this was and is unclear. However, the consequence was that especially in dual phase martensite plus ferrite structures, excess surface relief was produced. This made the replica more difficult to examine. Examples of this are shown in Figures 9-12. Thus for difficult martensite plus ferrite structures, we prefer the rubber replication media, otherwise either replication media proved adequate.

7.0 SUMMARY

The foregoing discussion illustrates that while publication quality micrographs may not always be obtainable from replicas, replication can be used to non-destructively obtain much of the information available by conventional bright field metallography. In applications for DND, it can be used to determine the phases present in, and the grain size of, steels which can be etched with nital. The techniques presented here can be easily adapted to other alloys, though different etchants are required. Coupled with knowledge

of hardness, which can be obtained using a portable hardness tester, and composition, which can also be determined in situ, replication can be used, in many cases, to determine if the steel used in a certain application is of the correct type and heat treatment, without destructive testing. Such determinations are commonly required after refits, after damage by excess heat from some source, and after a component of a widely used type fails as a result of incorrect microstructure. Such problems usually require only identification of the phases present and perhaps the grain size. These are relatively straightforward with the techniques developed in this work. If required for important investigations on high value components, subtler microstructural features, volume fraction ferrite in martensite for example, can also be examined. This might, for example, be required after extensive fire damage. Of special significance in failure analysis, the technology can be used to locate, relative to microstructural features, crack morphology in all the microstructures considered. In warranty disputes, the replication method can be used to assess such variables as heat treatment non destructively, before a problem component is returned to its supplier.

There are two limitations to replication. First and most important, if its non-destructive character is to be preserved, it is limited to examination of a near surface region. The depth of damage which can be tolerated is determined by the amount of material which can be removed by grinding, without making the component unfit for service. Normally this allows non-representative microstructures caused by surface damage, carburization or decarburization, and other effects to be removed. However, one must be convinced of this before data from replication can be used for engineering decision making. Where there is doubt the removal of specimens is required. The second problem with replication was identified earlier. Only microstructural features which either have relief themselves, such as cracks, or features which can be etched to have relief can be replicated. Consequently, alloys for which relief etching is inefficient will be difficult to examine with the technique.

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9.0 FIGURES

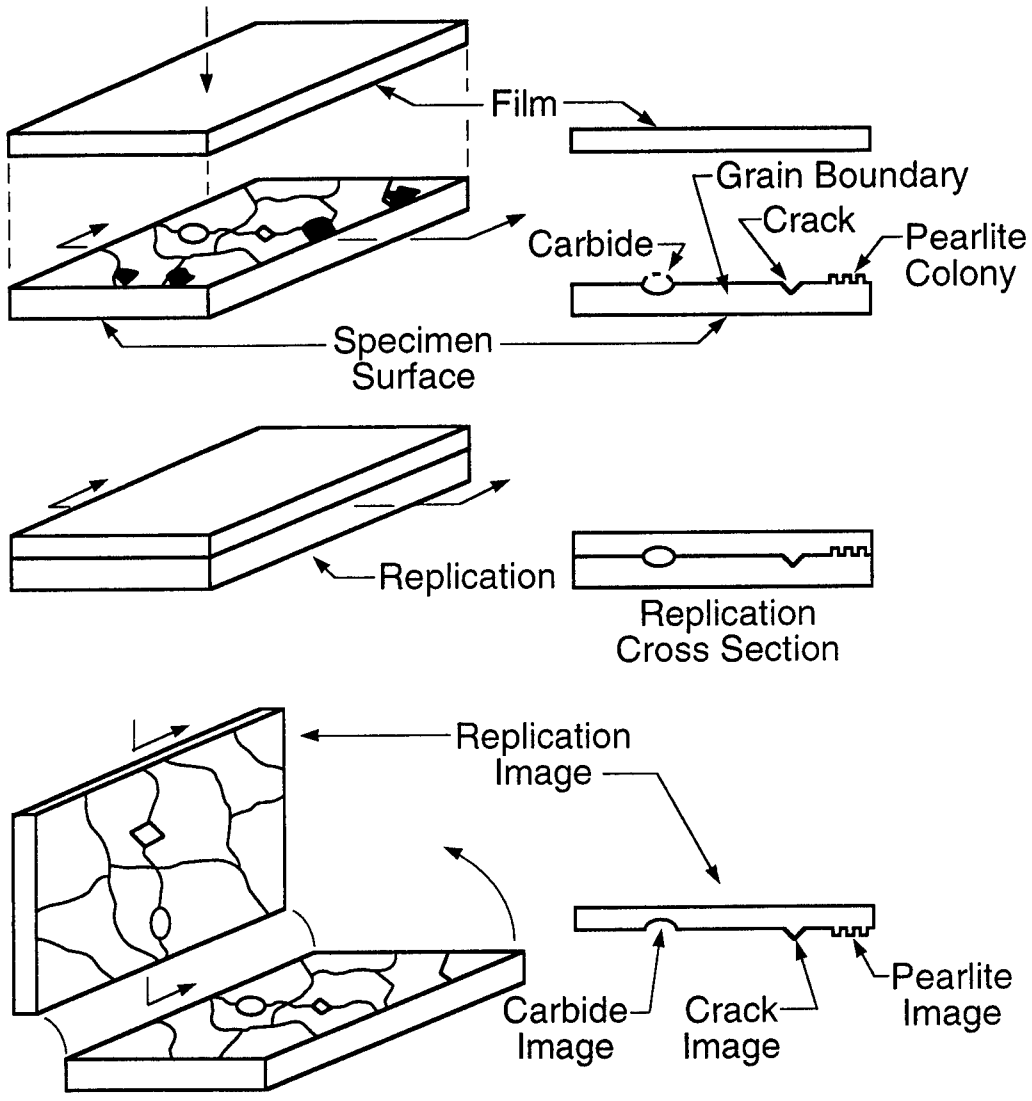

Figure 1. The Replication Process [After Nee, 1989].

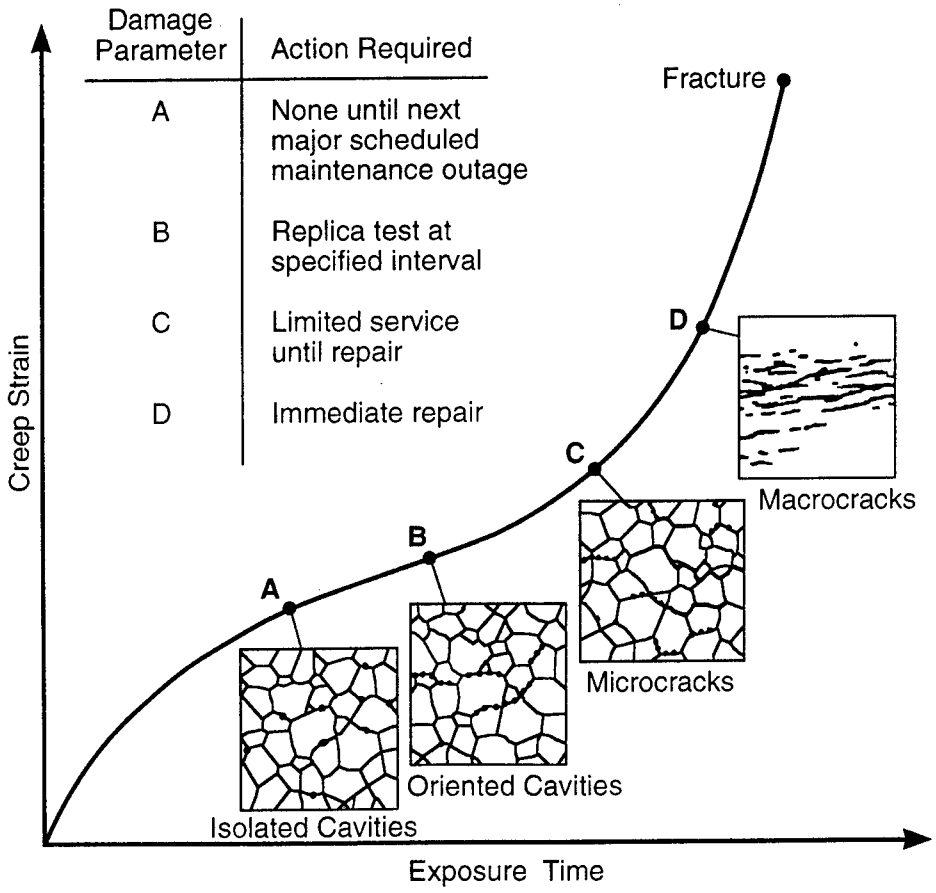

Figure 2. Development of Creep Cavities with Strain and Time [After Viswanathan, 1989].

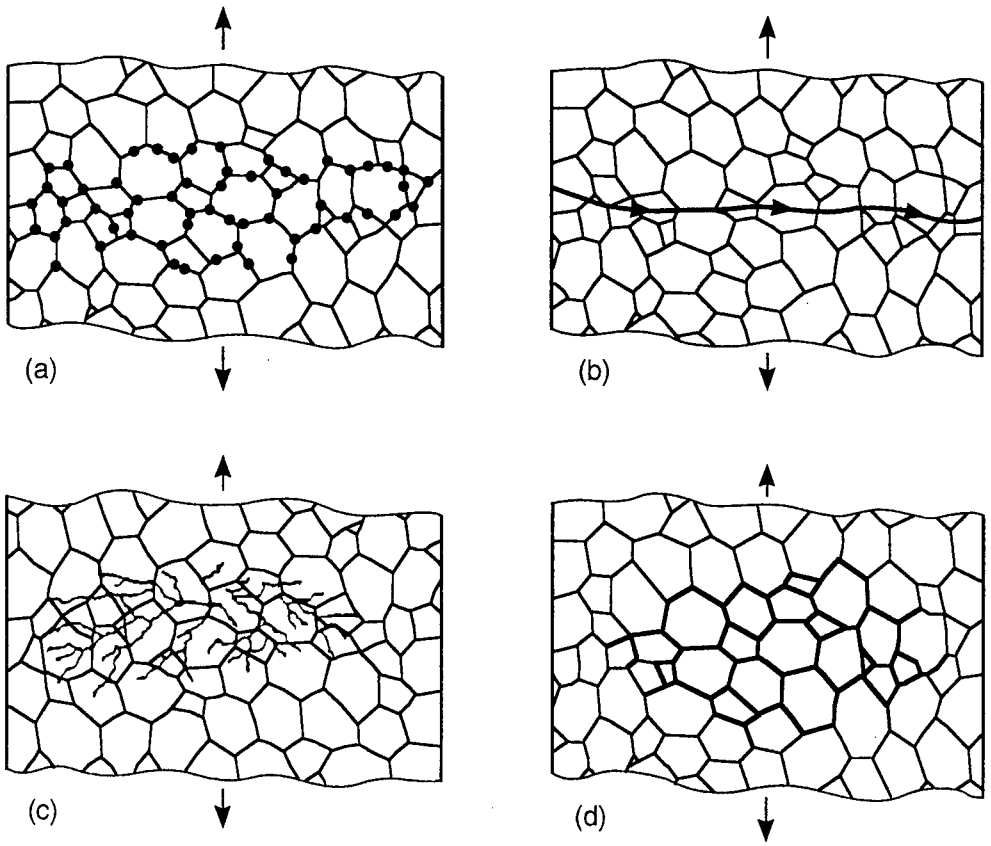

Figure 3. Crack types and their characteristic patterns. (a) Creep. (b) Fatigue.(c) Stress Corrosion. (d) Intergranular Corrosion [After Ludwigsen, 1987].

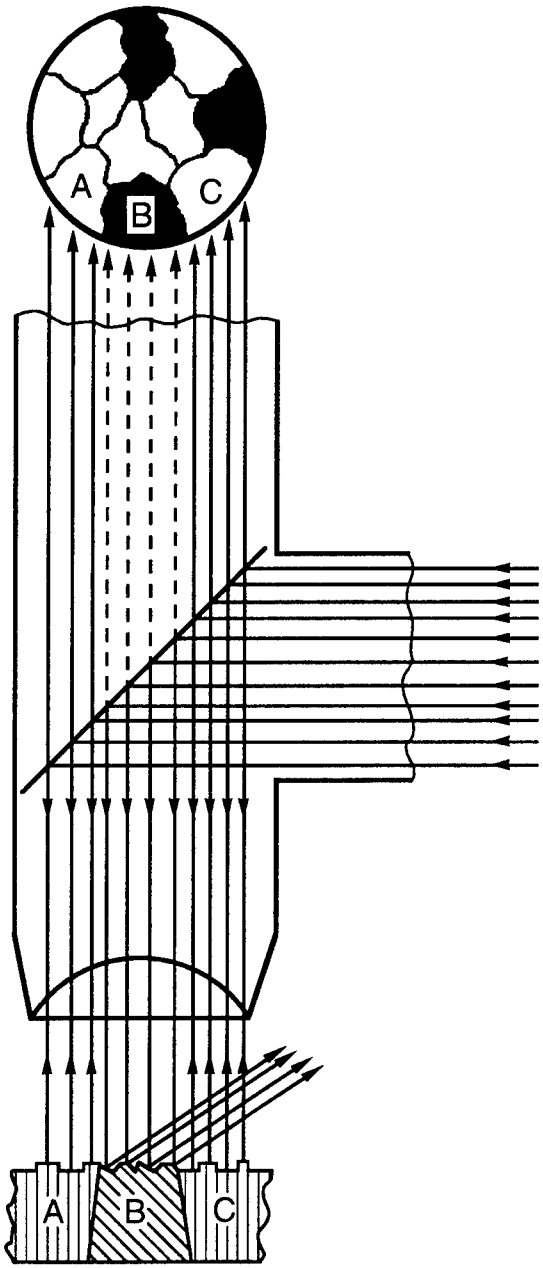

Figure 4. Oriented-Grain Luster produced by Grain Contrast Etching [After Kehl, 1949].

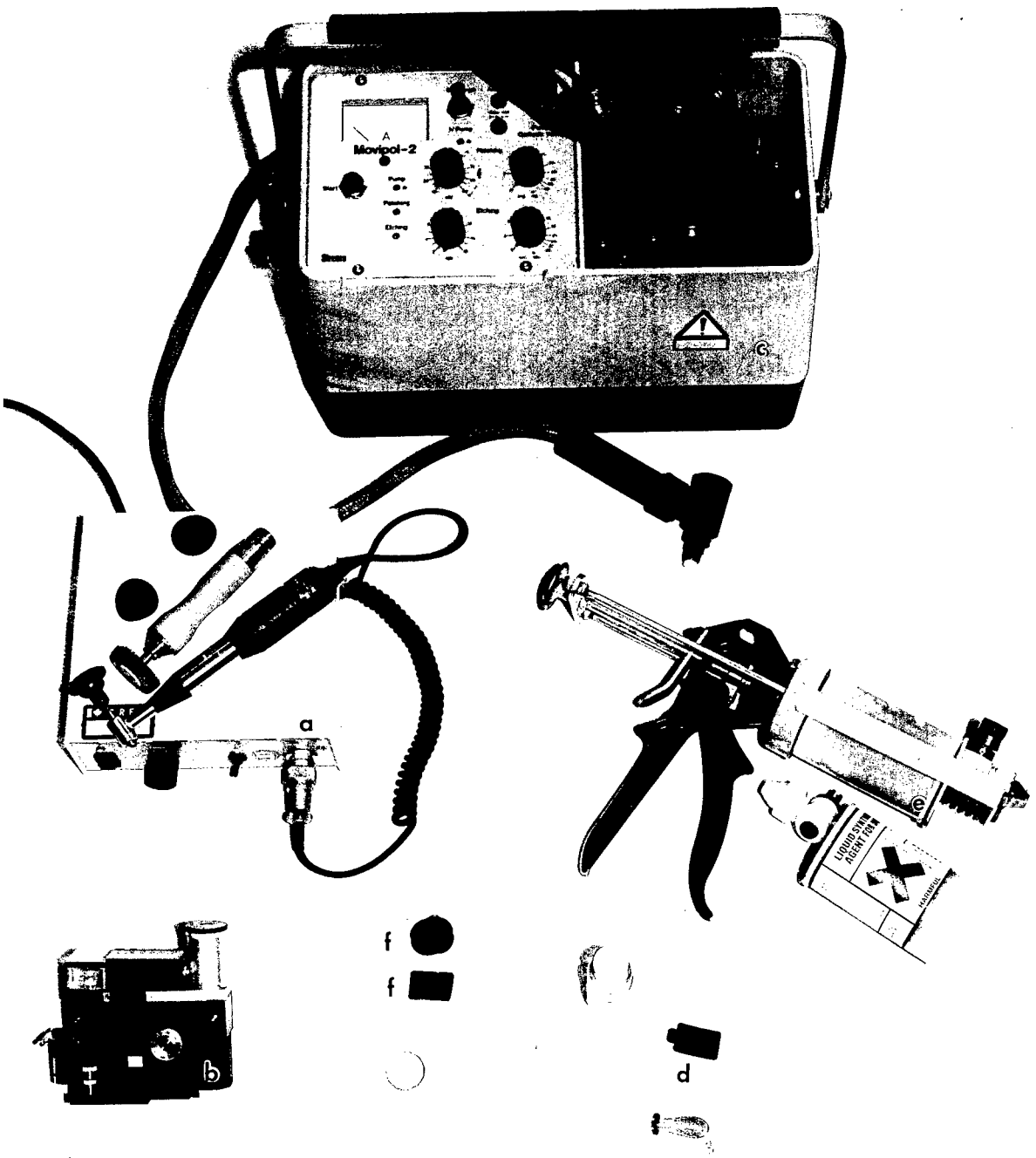

Figure 5. Field Metallography equipment including a hand grinder/polisher(a); a battery powered portable metallurgical microscope(b); an etcher-electropolisher(c), a Transcopy replication kit(d) containing foils, glass slides, ethyl acetate solvent, and a pipette; and an application gun for the two part rubber used in the Microset replication kit(e). Also shown(f), are replicas made with each system.

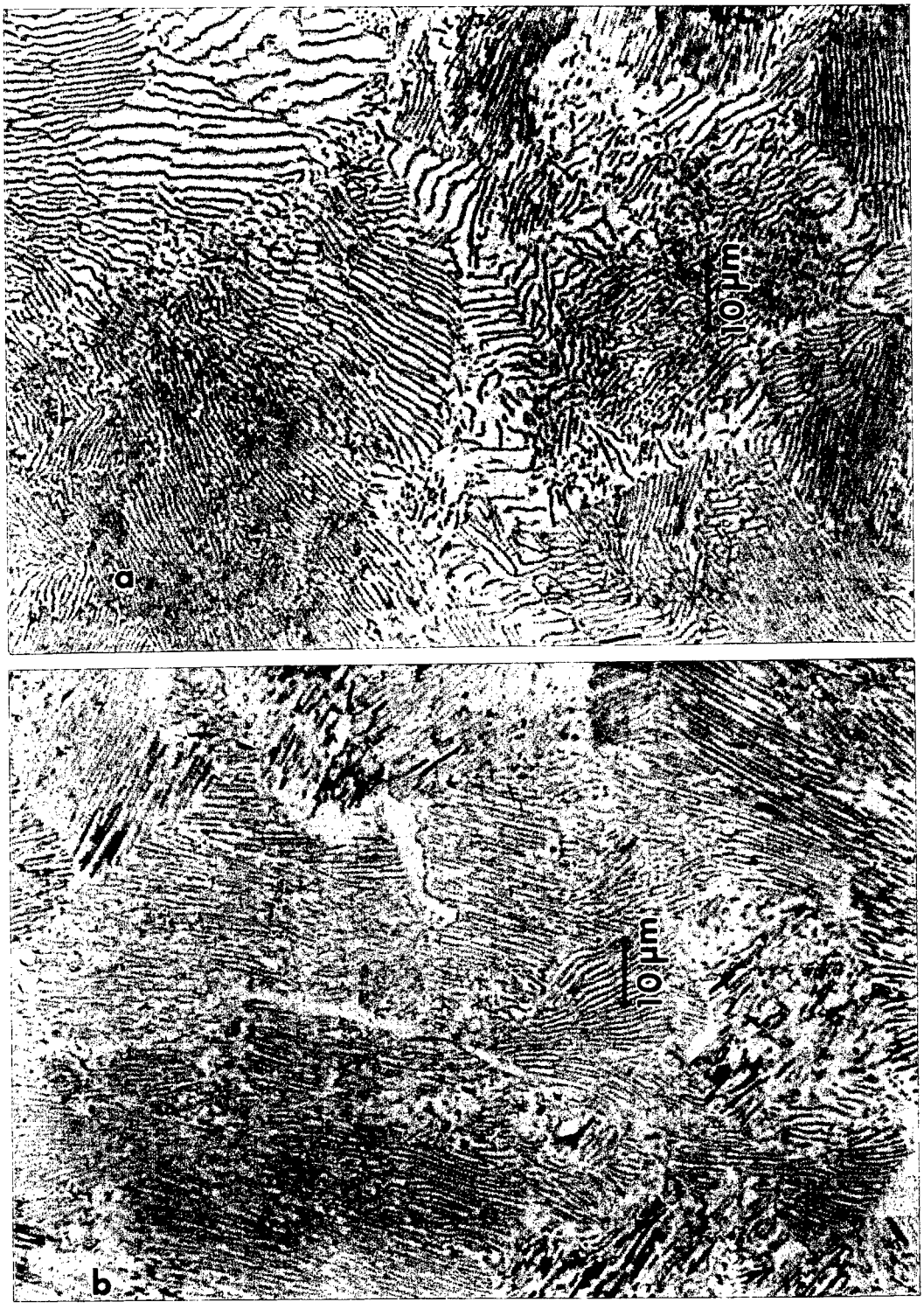

Figure 6. Micrographs of 1080 Steel showing its Pearlitic microstructure(2 % Nital etch). The micrograph of the conventional specimen(a) is very similar to that of the cellulose acetate replica(b).

Figure 7. Micrographs of ASTM A210 grade A-1 steel, showing its ferrite plus fine pearlite microstructure(2% nital etch). Though the pearlite lamellae could not be resolved, the contrast difference between the two phases, which is seen most clearly in the conventional micrograph(a), was well duplicated by both the cellulose acetate(b) and rubber replicas(c-next page).

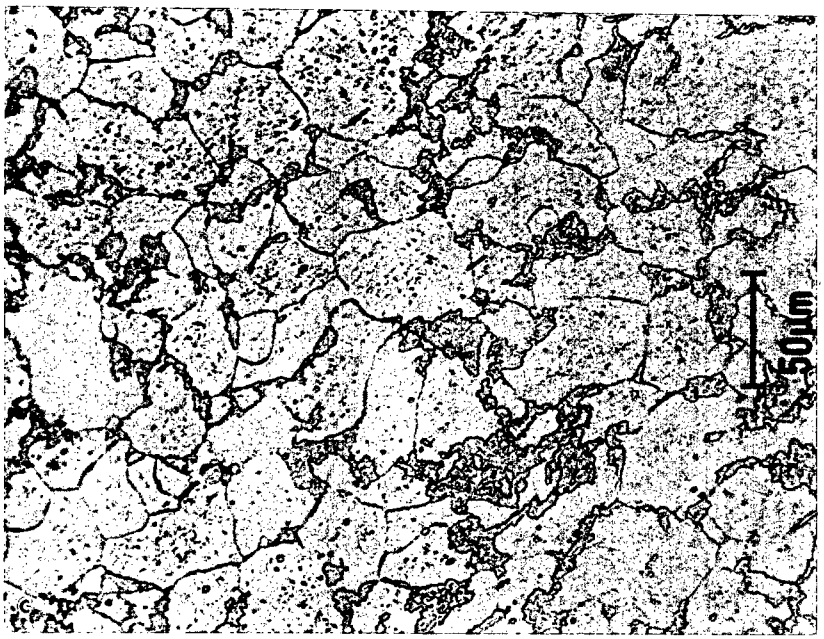

Figure 7 (continued). Micrographs of ASTM A210 grade A-1 steel. c. Rubber replica.

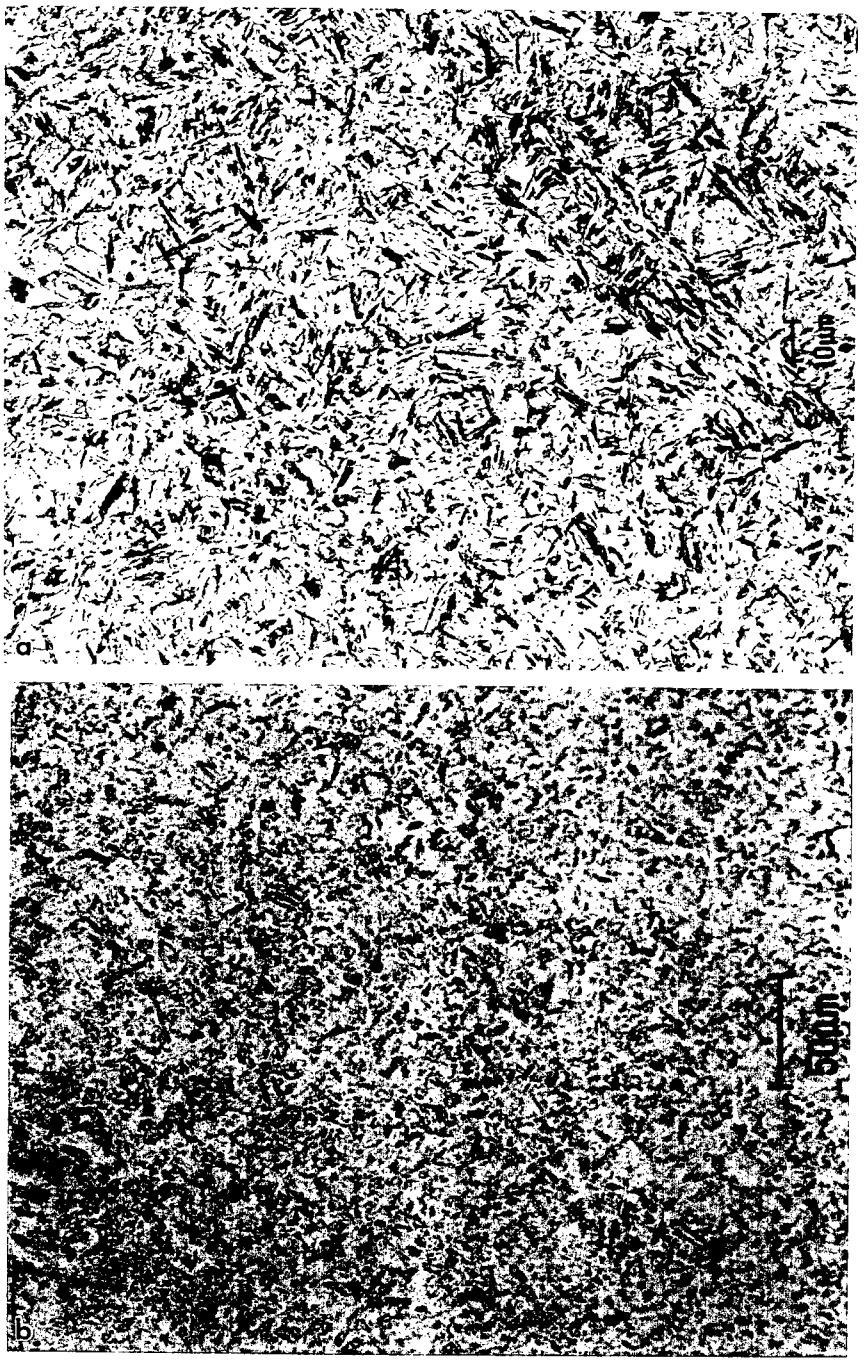

Figure 8. Micrographs of AISI 4340 steel water quenched from 870°C. Its lath martensitic microstructure is clearest in the conventional micrograph(a), but can also be seen, with a decrease in image contrast and sharpness, in the cellulose acetate(b) and rubber replicas(c-next page).

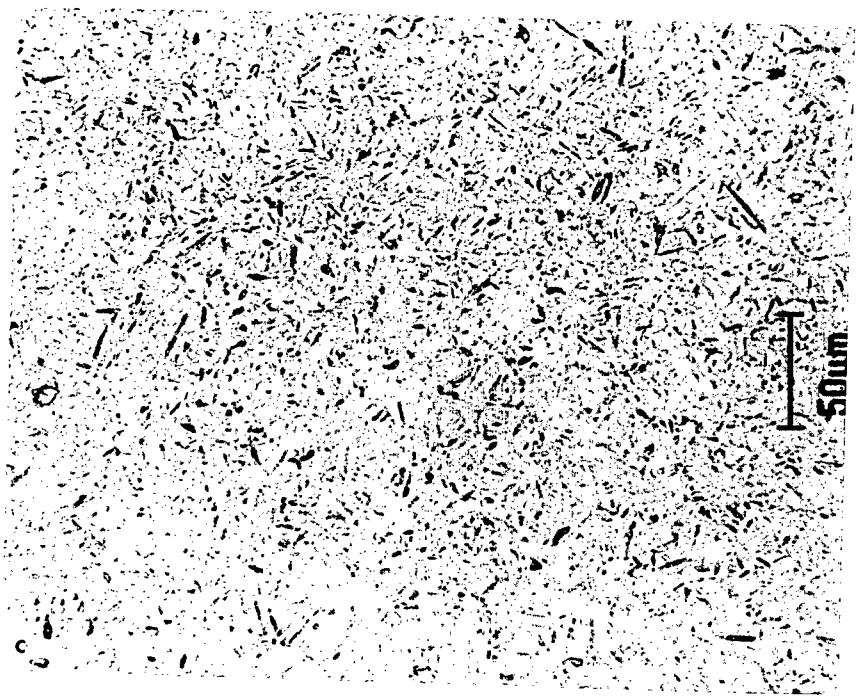

Figure 8 (continued). Micrographs of AISI 4340 steel water quenched from 870^{o}C. c. Rubber replica.

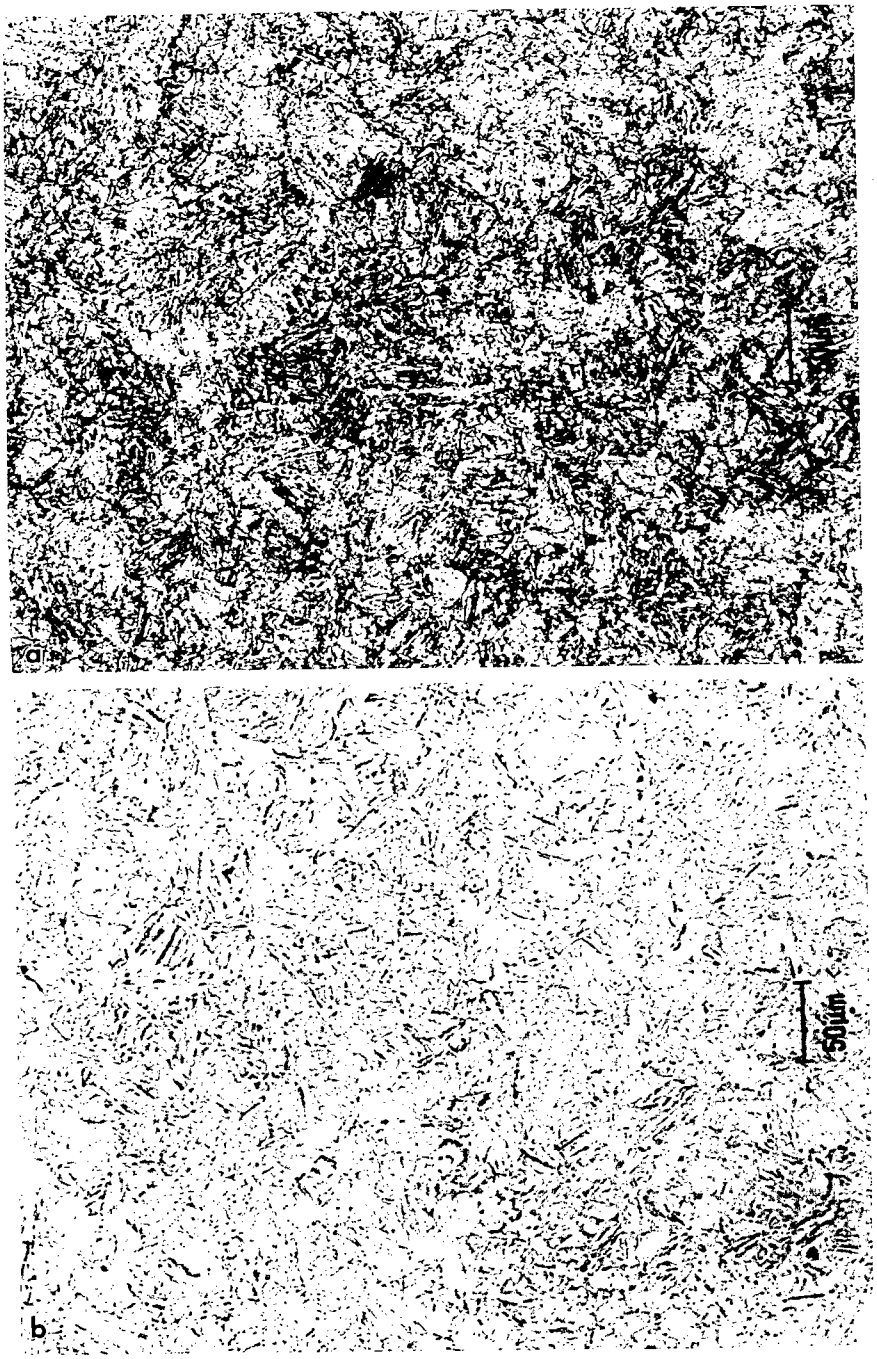

Figure 9. Micrographs of HY 80 steel, with a quench and tempered lath martensite structure. Details in the conventional micrograph(a) are acceptably reproduced by both the cellulose acetate(b) and rubber (c-next page) replicas.

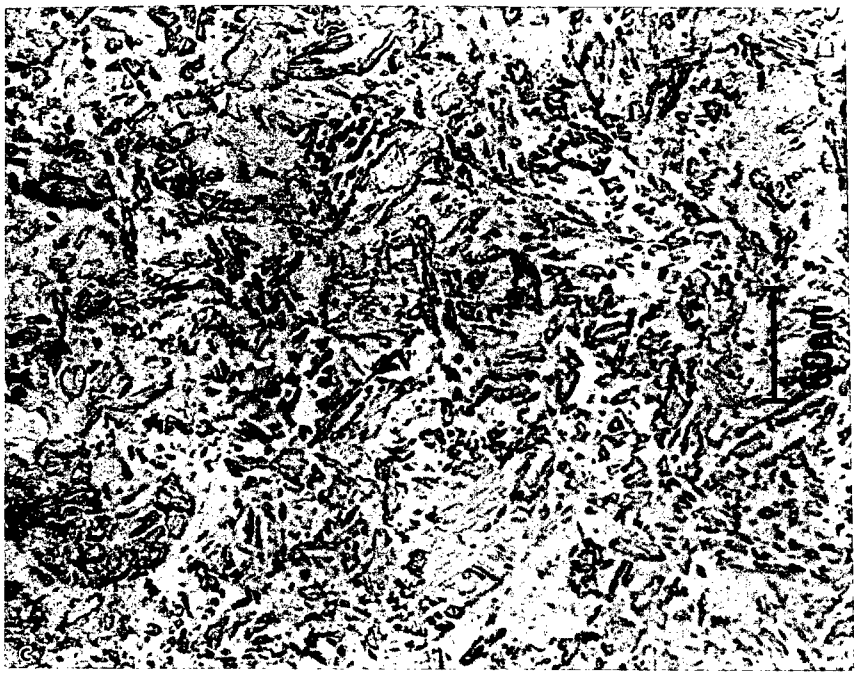

Figure 9 (continued). Micrographs of HY 80 steel. c. Rubber replica.

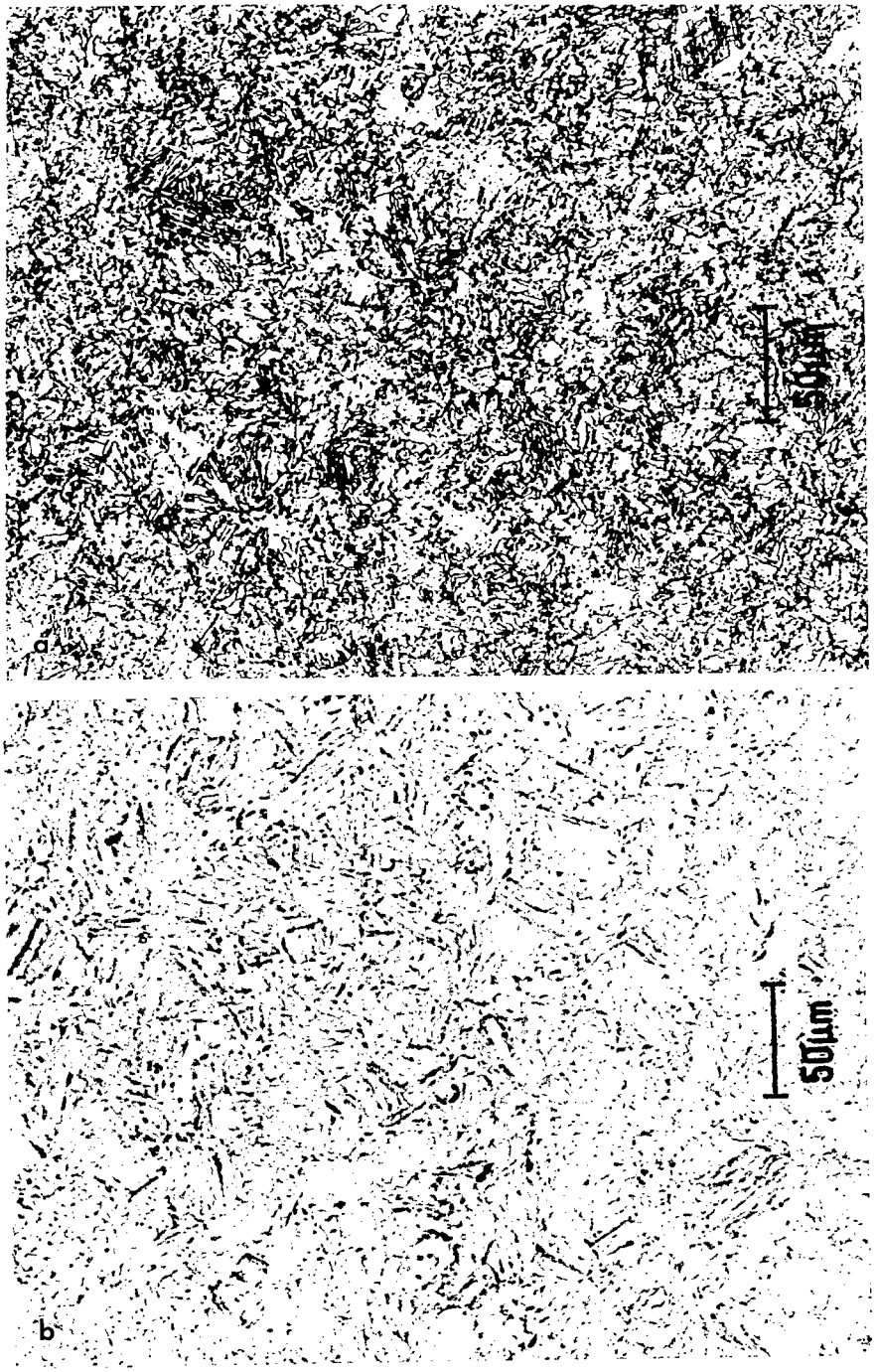

Figure 10. Micrographs of HY 100 steel, with a quench and tempered lath martensite structure. Details in the conventional micrograph(a) are adequately reproduced by both the cellulose acetate(b) and rubber replicas(c-next page).

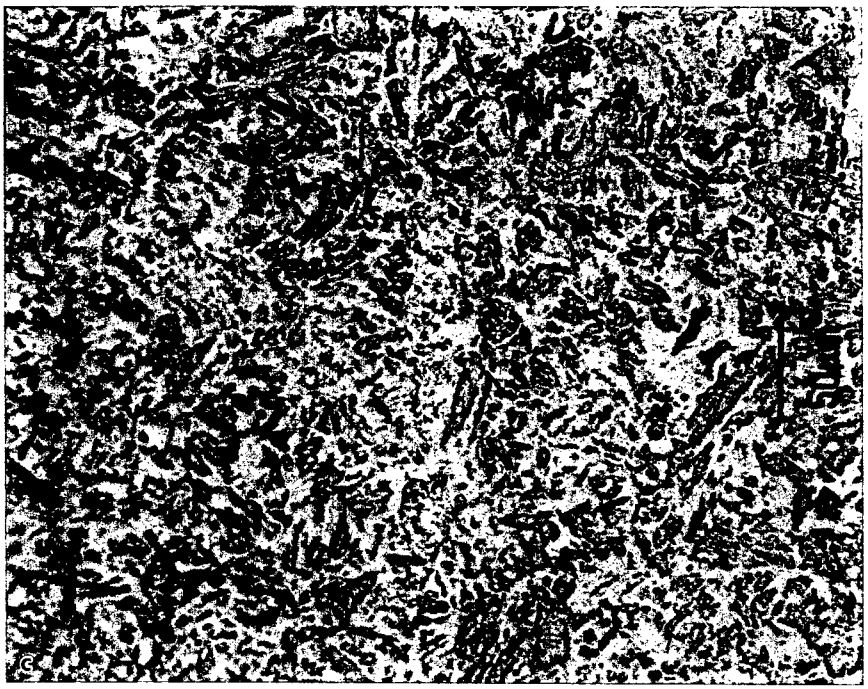

Figure 10 (continued). Micrographs of HY 100 steel, with a quench and tempered lath martensite structure. c. Rubber replica.

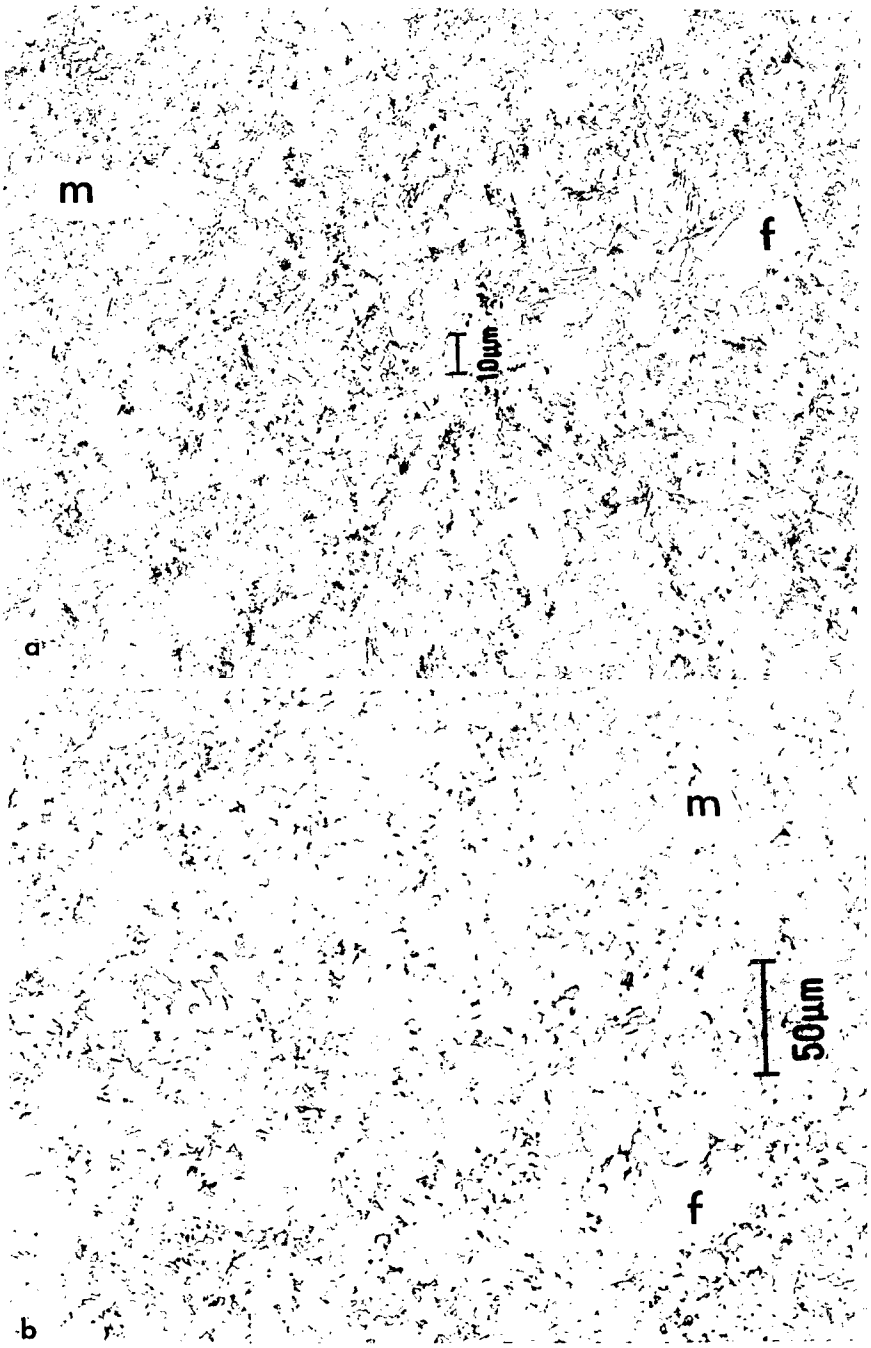

Figure 11. Micrographs of 4340 steel water quenched from roughly 780°C to give a martensite(m) plus ferrite(f) microstructure (2% nital etch). a. Conventional Micrograph. b. Cellulose acetate replica produced with an etching time roughly double that used for the conventional micrograph (a). (continued next page)

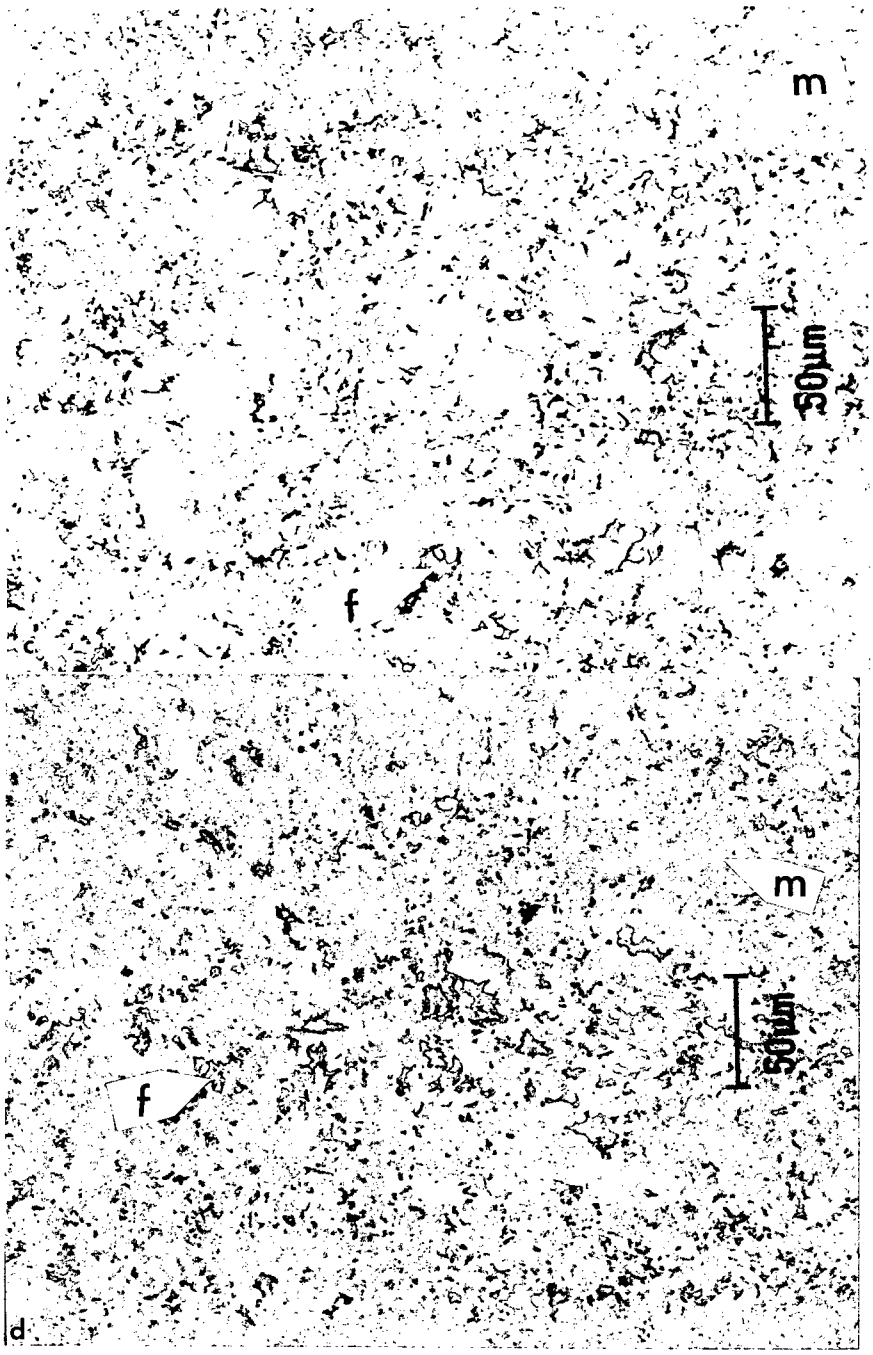

Figure 11 (continued). Micrographs of 4340 steel water quenched from roughly 780°C to give a martensite(m) plus ferrite(f) microstructure (2% nital etch). c. Cellulose acetate replica produced with an etching time four times that used to produce the conventional micrograph in (a). d. Cellulose acetate replica produced after etching for a longer time than used for (c).

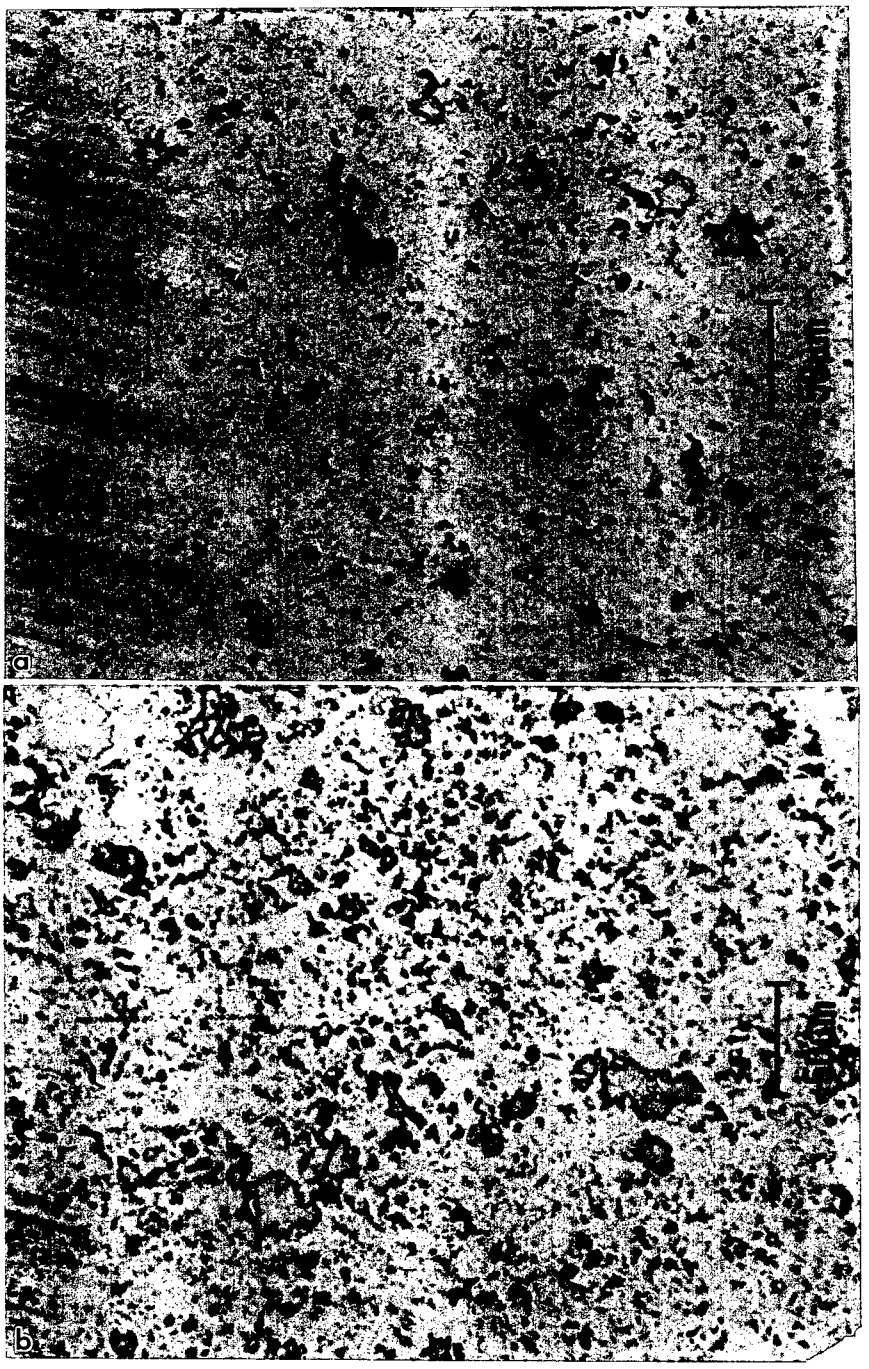

Figure 12. Micrographs of rubber replicas of 4340 steel water quenched from roughly 780°C to give a martensite(m) plus ferrite(f) microstructure (2% nital etch). a. Etching time 20 % longer than used for conventional metallography (Figure 11a). b. Longer etching time than for (a).

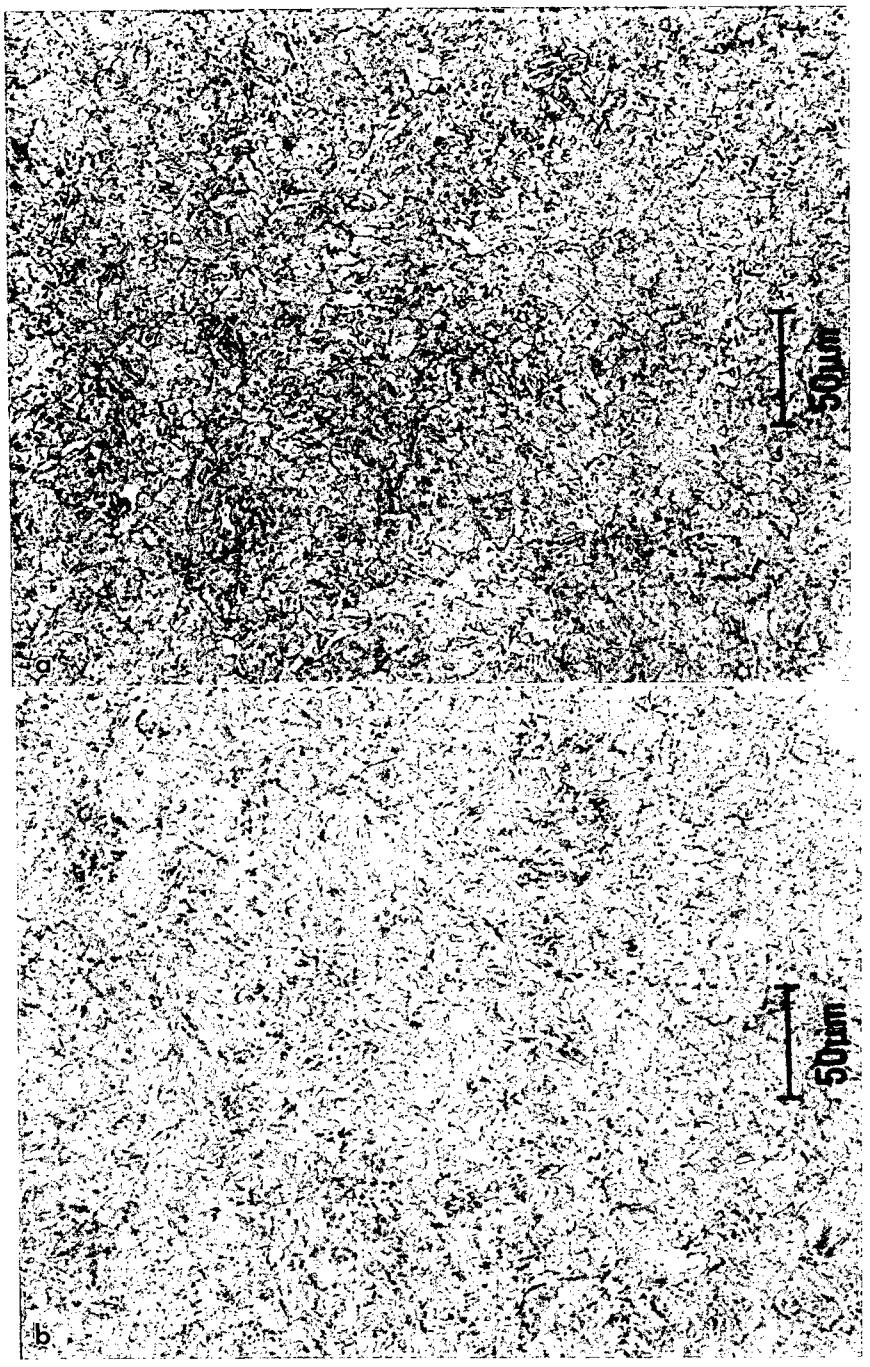

Figure 13. Microstructure of ASTM A336 steel with a (tempered?) martensite and ferrite microstructure resulting from an incorrect repair procedure. a. Conventional specimen etched with 2% Nital. b. Conventional specimen etched with sodium metabisulfate tint etchant. c (next page). Rubber replica from specimen etched with 2 % nital.

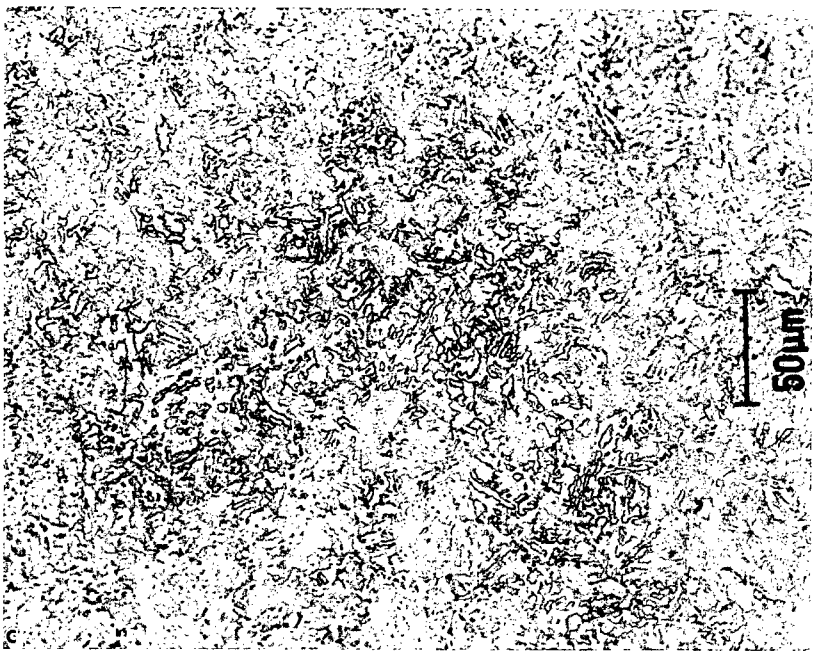

Figure 13 (continued). Microstructure of ASTM A336 steel with a (tempered?) martensite and ferrite microstructure resulting from an incorrect repair procedure. c. Rubber replica from specimen etched with 2 % nital.

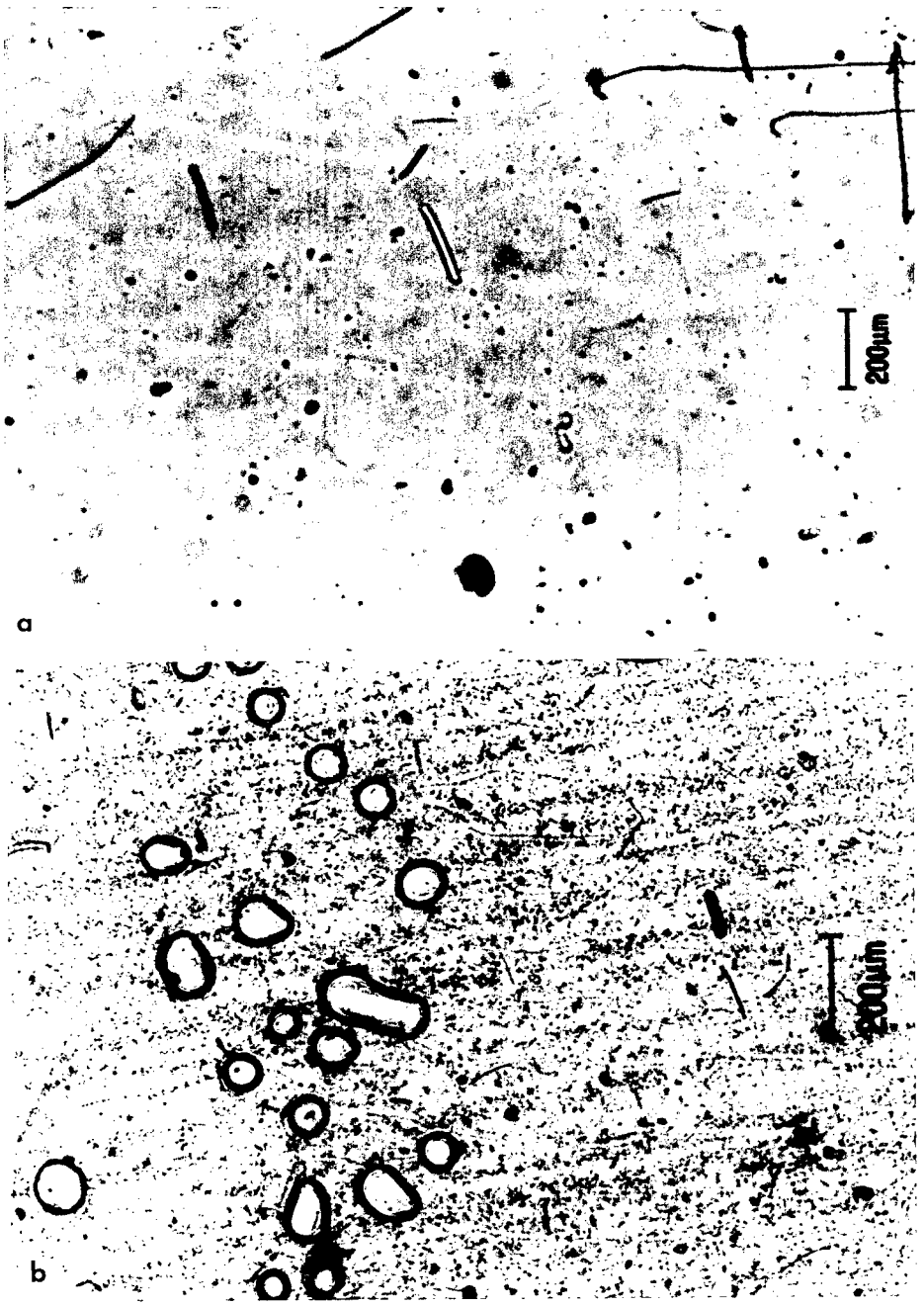

Figure 14. Common artifacts in replica foils. Dirt(a) occurs at the same plane of focus as the replica surface while bubbles(b) occur at a slightly different plane.

ANNEX A

PROCEDURES FOR GRINDING, POLISHING, ETCHING, AND REPLICATION

The procedures used to produce the replicas shown in the report were:

1. **Rough grinding** to a 100 grit finish using a body sander or hand grinder. Care was used not to overheat the surface. The surface being ground was always cool enough that one could put their hand on it.

2. **Finish grinding** the surface was done with the portable grinding/polishing unit. A speed setting between 6 and 9 was used. Progresively finer grit (180, 240, 320, 400 and 600 grit SiC) grinding papers were used. At each grit size, grinding was continued until no scratches caused by the previous step could be seen on surface with the portable optical microscope. For each grinding step, this normally required about four minutes per square inch. Each grinding step was done so that the scratches from it were at 90° to those of the previous step. Also between each grinding step, debris which might cause scratching was removed by washing with soap and water and drying with a clean soft cloth.

3. **Cleaning** the surface was done prior to polishing, and between each polishing step to remove any debris, grit, or polishing compound from the area of interest. This was done by washing with dish soap and water soaked cotton balls, rinsing with hot water, then rinsing with methanol or ethanol and then drying the surface with a hot air gun.

4. **Polishing** was done with the portable hand grinder, using, first, a six micron diamond paste on a napless polishing cloth at a speed setting of 5, then a three micron diamond paste on a napped cloth at a speed setting of 4 and finally a one micron paste on a napped cloth at a speed setting of 3. In each case, odorless kerosene or other appropriate cutting oil was occasionally added to the polished surface as a polishing fluid, and polishing was continued until examination with the optical microscope showed all scratches from the previous grit size were just removed. This normally took about four minutes per square inch for each paste. Normally about one square inch was polished.

5. **Etching** was done with the portable electropolisher/etcher using one of two methods depending on the material and the information sought. Both methods used the capability of the electropolisher/etcher to etch, for a set amount of time a roughly 6 mm diameter spot on the polished surface, without damaging the surrounding region. The first method, which was most useful for pearlitic, pearlite plus ferrite and martensitic steels, involved etching the surface until the luster of the polished metal surface just started to dull. The surface was then examined with the portable optical microscope, to determine if etching had revealed the features of interest. If it had not, adjustments to the etching time were made. If it had, shorter and longer (by an amount based on experience, with 20 to 50%

changes being a reasonable starting point) times were used to etch two other spots adjacent to the first. The purpose of this last step was to produce the best possible replica, containing details not observable with the portable microscope, for examination with a higher quality metallographic microscope in the laboratory. The second method, which was useful for steels containing phases such as martensite and ferrite with significantly different etching rates, required more work. This method, incremental etching, involved etching the same 6 mm spot several times, using times determined by experimentation to be appropriate for each phase and replicating after each etching cycle. Scribed marks were used so that the same region could be relocated each time.

6. **Replication with cellulose acetate replicas** from the Transcopy replication kit involved:

a. softening a cellulose acetate foil with 6 to 7 drops of Transcopy fluid(ethyl acetate);

b. rolling this liquid around the film to wet it completely;

c. after 20 seconds pouring off any remaining liquid;

d. immediately applying this foil to the etched surface by pressing slowly from one edge to the other, taking care not to adjust the replica position after it was pressed into place;

e. allowing the replica to harden for two minutes;

f. removing the replica by pulling slowly on the tab with a firm steady force, stopping and checking to see if the replica was still wet; and

g. if necessary, allowing the still attached part of the replica more time to dry if the part removed was still wet, then repeating step f.

Once removed, the replica foil was mounted on a microscope slide with its adhesive backing, as well as tape around the replica edges.

7. **Replication with rubber replicas** was somewhat simpler than the procedure just described. First the surface was treated with proprietary wetting agent, then the rubber, premixed with catalyst by the application system, was applied to the surface and spread evenly. After hardening for 10 minutes on the surface, the replica was removed and mounted on a glass side with double sided tape.

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