INIS.
·MRN AU8105592

AAEC/E513

AAEC/E513

AUSTRALIAN ATOMIC ENERGY COMMISSION
RESEARCH ESTABLISHMENT
LUCAS HEIGHTS

THE OPTICAL METALLOGRAPHY OF SOME MARAGING STEELS

by

E. MELLER
J.T.A. POLLOCK

May 1981

ISBN 0 642 59711 1

Attention Microfiche User,

The original document from which this microfiche was made was found to contain some imperfection or imperfections that reduce full comprehension of some of the text despite the good technical quality of the microfiche itself. The imperfections may be:

- missing or illegible pages/figures
- wrong pagination
- poor overall printing quality, etc.

We normally refuse to microfiche such a document and request a replacement document (or pages) from the National INIS Centre concerned. However, our experience shows that many months pass before such documents are replaced. Sometimes the Centre is not able to supply a better copy or, in some cases, the pages that were supposed to be missing correspond to a wrong pagination only. We feel that it is better to proceed with distributing the microfiche made of these documents than to withhold them till the imperfections are removed. If the removals are subsequestly made then replacement microfiche can be issued. In line with this approach then, our specific practice for microfiching documents with imperfections is as follows:

1. A microfiche of an imperfect document will be marked with a special symbol (black circle) on the left of the title. This symbol will appear on all masters and copies of the document (1st fiche and trailer fiches) even if the imperfection is on one fiche of the report only.

2. If imperfection is not too general the reason will be specified on a sheet such as this, in the space below.

3. The microfiche will be considered as temporary, but sold at the normal price. Replacements, if they can be issued, will be available for purchase at the regular price.

4. A new document will be requested from the supplying Centre.

5. If the Centre can supply the necessary pages/document a new master fiche will be made to permit production of any replacement microfiche that may be requested.

The original document from which this microfiche has been prepared has these imperfections:

☒ missing pages/~~figures~~ numbered: 3 not printed

☐ wrong pagination

☐ poor overall printing quality

☐ combinations of the above

☐ other

INIS Clearinghouse
IAEA
P. O. Box 100
A-1400, Vienna, Austria

AUSTRALIAN ATOMIC ENERGY COMMISSION
RESEARCH ESTABLISHMENT
LUCAS HEIGHTS

THE OPTICAL METALLOGRAPHY OF SOME MARAGING STEELS

by

E. MELLER
J.T.A. POLLOCK

ABSTRACT

The structural features of maraging steels, including those that impose limitations on strength and toughness, are briefly reviewed. Phase relationships in the iron-nickel matrix system are introduced which, in conjunction with alloy composition and heat treatment, allow the interpretation of microstructure. Details are presented of methods for revealing the microstructure of maraging steels; the prior austenite grain size following a variety of heat treatments is of special interest since excessive grain size can lead to poor ductility. These methods are illustrated with micrographs of the microstructures of MAR 350 and MAR 400 steels in the cast, solution-treated, aged and cold-worked states.

National Library of Australia card number and ISBN 0 642 59711 1

The following descriptors have been selected from the INIS Thesaurus to describe the subject content of this report for information retrieval purposes. For further details please refer to IAEA-INIS-12 (INIS: Manual for Indexing) and IAEA-INIS-13 (INIS: Thesaurus) published in Vienna by the International Atomic Energy Agency.

MARAGING STEELS; METALLOGRAPHY; MICROSTRUCTURE; PHASE STUDIES; GRAIN SIZE; MECHANICAL PROPERTIES; HEAT TREATMENTS

CONTENTS

1. INTRODUCTION

The mechanical properties of maraging steels make them suitable materials for the construction of highly stressed, lightweight components. In a research program on centrifuge enrichment of uranium, these steels have been broadly assessed during the past few years. Microstructural studies have been a significant part of this assessment since acceptable combinations of strength, toughness and ductility are dependent on structure. The primary goal of these microstructural studies is to assess the effect of heat treatments. In particular, the prior austenite grain size must be carefully controlled as excessive grain size causes poor ductility [Nakazawa et al. 1978] and the presence of inclusions or unwanted precipitates leads to a reduction in toughness [Magnee et al. 1973]. In addition, poor homogeneity, resulting from inadequate melting procedures or thermomechanical processing, may be detected by metallographic examination and corrected before mechanical processing.

It was found that experimental details available in the literature were inadequate for the successful and reliable preparation of surfaces suitable for metallographic examination. On occasion, metallographic examination failed to reveal details identified by transmission electron microscopy (TEM) that were sufficiently large for optical resolution. As a consequence, the AAEC has developed improved techniques for the optical metallography of maraging steels to complement TEM studies carried out at this laboratory [Warren et al. 1980].

This report deals specifically with the metallography of MAR 350 and MAR 400* steels. A brief review of the physical metallurgy of maraging steels is presented; this includes details of grade compositions and mechanical properties. Metallographic procedures used to reveal microstructural details of maraging steels in various conditions are then discussed. Particular attention is given to the prior austenite grain size as a means of detecting, and hence controlling, recrystallisation and grain growth during heat treatment.

---

* The serials 350 and 400 denote the ultimate tensile strengths of the steels in the old KSI (lb in^{-2} x 10^{3}) units and are the accepted grade designations.

2. PHYSICAL METALLURGY

Maraging steels are characterised by very low carbon contents and the use of substitutional alloying elements in the iron-nickel matrix. The former allow an easily deformed martensite to form on cooling from the austenite region of the iron-nickel diagram, and the latter produce age-hardening at low temperatures where, because of thermal hysteresis, there is no significant reversion to austenite. Although maraging steels are susceptible to stress corrosion and hydrogen embrittlement, they compare favourably with other non-stainless, quenched and tempered carbon alloy steels and are tougher at the same strength level.

Chemical compositions of typical maraging steels in the iron-nickel-cobalt-molybdenum group are presented in Table 1, together with maximum attainable fracture stress, elongation at fracture and fracture toughness values. It should be noted that although attainable strength levels depend on alloying content, strengths achieved in practice are governed by heat treatment. Hence, there is considerable overlap of mechanical properties among these steels.

TABLE 1

TYPICAL CHEMICAL COMPOSITIONS AND MECHANICAL PROPERTIES OF MARAGING STEELS

Steel Grade	Nominal Composition (wt %)							Ultimate Tensile Strength (MN m^{-2})	% Elongation at Fracture	Fracture Toughness K_{IC}, MN $m^{-3/2}$
	Fe	Ni	Co	Mo	Ti	Al	C			
MAR 250	Bal*	18	8	5	0.4	0.1	<0.03	1750	13	132
MAR 300	Bal	18	9	5	0.6	0.1	<0.03	1960	11	99
MAR 350	Bal	17	12	4	1.5	0.1	<0.03	2450	9	50
MAR 400	Bal	13	15	10	0.2	0.1	<0.03	2700	5	40

*Bal = balance of nominal composition

The maraging characteristics are derived from the phase relationships obtained at the iron-rich end of the iron-nickel phase diagram. Two phase diagrams, the equilibrium [Owen and Humphrey 1949] and the metastable [Jones and Liu 1949], must be considered. The equilibrium diagram shown in Figure 1a reveals that for alloys containing more than about 7 per cent nickel, the

(iv) the chemical inertness of the mounting material in perchloric acid solutions.

Two thermosetting compounds, bakelite and acrylic, and two cold setting compounds, epoxy resin and acrylite, were considered. The acrylic and acrylite compounds could not be used owing to their reaction with perchloric acid solution. Although edge retention with epoxy resin is not entirely satisfactory, the setting compound was chosen in preference to bakelite because the latter is a potential explosive hazard when used in perchloric-based electrolytes.

3.2 Grinding and Rough Polishing

3.2.1 Grinding

Mounted samples were hand ground on No.120, 320, 800 and 1200 silicon carbide abrasive papers, with running water as a lubricant. Some 0.25 mm thick strip samples were ground using only No. 800 and 1200 papers. A minimum of two 90° changes in grinding direction was carried out for every grade of abrasive paper.

3.2.2 Rough mechanical polishing

Rough polishing was carried out on a rotating napless Terylene cloth using 4 to 8 μm diamond paste, with kerosene as lubricant. Specimens were hand held and rotated counter to the wheel rotation. Microscopy was used to determine the end point of rough polishing.

> Note: Mechanical polishing using finer grades of diamond paste was completely unsatisfactory. Relief artefacts, caused by deformation resulting from rolling or preliminary grinding and rough polishing, formed on all samples and prolonged polishing only increased their size.

3.2.3 Rough chemical polishing

Specimens in the as-ground or rough mechanically polished condition were chemically polished by immersion and/or swabbing in a 5 per cent HF (48 per cent conc.) 45 per cent H_2O_2, 50 per cent H_2O solution for 5 to 20 seconds at room temperature. The surface finish often revealed some structural detail, but the main role of this polishing step was the removal of prior deformation due to grinding. Chemical polishing after mechanical polishing has proved the best base for electropolishing.

3.3 Electrolytic Final Polishing

The most satisfactory final surface polish was obtained by electrolytic methods, using either the simple 'beaker technique' or a proprietary apparatus called Visapol. Both methods use electrolytes based on perchloric acid.

3.3.1 Beaker technique

The electrolyte was a five per cent $HClO_4$ in methanol solution at -76°C. A quantity of this electrolyte was chilled in a cathodic stainless steel beaker surrounded by a mixture of dry ice and acetone. After connecting a heavy gauge copper wire handle/conductor, the sample was chilled in three stages before immersion in the cooled electrolyte. This chilling sequence was necessary to prevent fragmentation of the epoxy resin mount owing to thermal shock. When the sample was at the electrolyte temperature, it was momentarily withdrawn, connected positively to the power pack and re-immersed in the electrolyte at a potential of 65 V d.c. After 30 seconds of vigorous stirring, the sample was removed, warmed to room temperature by reversing the chilling sequence and, after rinsing in ethanol, dried and examined by microscopy.

3.3.2 The Visapol technique

A five per cent $HClO_4$ in glacial acetic acid electrolyte was used at room temperature. The major component of the Visapol equipment is a polishing cell fitted with a window to enable the operator to inspect and control the polishing process by viewing the sample surface with an optical microscope. The polishing unit is backed by an electrolyte control unit with facilities to alter the electrolyte flow rate and instantaneously change from electrolyte to a rinsing solution or to another electrolyte. A power pack is also available with this unit which may be mechanically programmed for automatic polishing and etching or for manual operation.

Although samples may be polished unmounted after a chemical polish, the best procedure is to mount them in epoxy resin and follow the rough polishing procedure outlined in Section 3.1. The sample is held onto the mask of the polishing cell by a conducting clamp and the surface viewed through the cell window using either an upright or an inverted microscope. A predetermined flow rate and a d.c. potential of 70 to 90 V are then applied and polishing carried out either automatically or under manual control until the end point is observed. It should be noted that with the Visapol method, cathode size and separation are fixed, leaving d.c. potential, electrolyte flow rate and time as variables.

3.3.3 Etching

No chemical etchant gave reproducible results. Electrolytic etching gave satisfactory results, but the heat treatment history of the sample determined both the choice of electrolyte and the degree of satisfaction. In particular, the etching behaviour of aged samples was difficult to predict and had to be approached empirically.

This method produced relief etching. In general, very light relief etching was satisfactory; however, normal bright field illumination is unsuitable for revealing the microstructural details and Nomarski type differentital interference contrast objectives were employed to enhance the low relief image.

Two etchants, ten per cent aqueous oxalic acid and ten per cent aqueous chromic acid, were used to 'beaker' etch solution-treated and/or cold-worked samples at about 10 V for times in excess of 10 minutes. However, with the Visapol unit, a one per cent hydrochloric acid in methylated spirit solution gave more superior results than the beaker technique with any etchant at a lower potential of about 2 V and shorted times of about 30 s. Aged samples could not be satisfactorily etched using the beaker method.

All aged samples were polished and etched in a five per cent perchloric acid in glacial acetic acid solution using the Visapol unit. Each sample was treated empirically, the quality of the result being dependent on the degree of ageing. Samples aged at 500°C were very difficult to prepare. These samples contained very high densities of precipitates which were metastable if the sample had been treated at 500°C for less than 20 hours. The surfaces of such samples were chemically very active and staining occurred rapidly in the absence of applied potential. After grinding and mechanical polishing, as outlined in Sections 3.2.1 and 3.2.2, these samples were electrolytically polished at 80 V for about 20 seconds, followed by etching for one second at two volts.

4. EXAMPLES OF PREPARED MICROSTRUCTURES

The results obtained by the beaker technique with unaged samples, although satisfactory, were considerably more time-consuming than those produced using the Visapol unit. In general, the Visapol results were uniformly better because the effective visual control over the polishing/etching cycle allowed the process to be stopped close to the optimum surface finish. For this reason, all of the micrographs described in this section are of surfaces prepared using the Visapol unit.

4.1 As-cast Samples

As-cast samples were prepared with the Visapol unit by electropolishing using five per cent perchloric acid in glacial acetic acid and electro-etching in a one per cent hydrochloric acid in methylated spirits solution. Occasionally, localised passivation, associated with large intermetallic inclusions, occurred during electrolytic polishing of as-cast samples. Examples of as-cast MAR 400 samples prepared in this manner are presented in Figures 2a and b. The initial inhomogeneous structure of the arc-melted material is clearly revealed in Figure 2a. After homogenising at 1150°C for 60 minutes, a large-grained lathe martensite structure is observed (Figure 2b).

4.2 Solution-treated and Worked Samples

Hot-rolled, cold-rolled and solution-treated examples of MAR 350 are shown in Figures 3a, b and c. These microstructures were prepared with the Visapol unit by electropolishing using five per cent perchloric acid in glacial acetic acid, followed by electro-etching in one per cent hydrochloric acid in methylated spirits. The prior austenite grain size is revealed in all samples including the as-received bar; in the latter case, the very large austenite grain size suggests that it was hot worked above 900°C.

4.3 Aged Samples

Aged samples were polished and etched in a five per cent perchloric acid in glacial acetic acid solution. Examples of MAR 350 and MAR 400 samples prepared in this manner are shown in Figures 4a, b and c. Figures 4a and b show microstructures of samples which had been aged at 500°C, the recommended ageing temperature. In each case, the prior austenite grain size is clearly revealed.

The coarse precipitate present in the MAR 400 sample aged at 865°C for 120 minutes (Figure 4c) was identified by TEM as a μ-phase [Warren et al. 1980]. This phase is thought to be the cause of poor fracture toughness in MAR 400 steels [Magnee et al. 1973]. Its presence usually results from inadequate solution treatment or slow cooling through the sensitising temperature range 750 to 900°C.

5. CONCLUSION

Metallographic procedures have been successfully used to reveal the microstructures of maraging steels, particularly the prior austenite grain size after various heat treatments.

6. REFERENCES

Jones, F.W. and Liu, Y.O. [1949] - J.Iron Steel Inst., 163:132.

Magnee, A., Viatour, P., Drapier, J.M., Coutsoupaois, D. and Habraken, L. [1973] - Cobalt, 1:3.

Nakazawa, K., Kawabe, Y. and Winneki, S. [1978] - Mater. Sci. Eng., 33:49.

Owen, E.A. and Humphrey, W.I. [1949] - J.Iron Steel Inst., 163:121.

Warren, R., Kelly, P.M. and Pollock, J.T.A. [1980] - AAEC/E483.

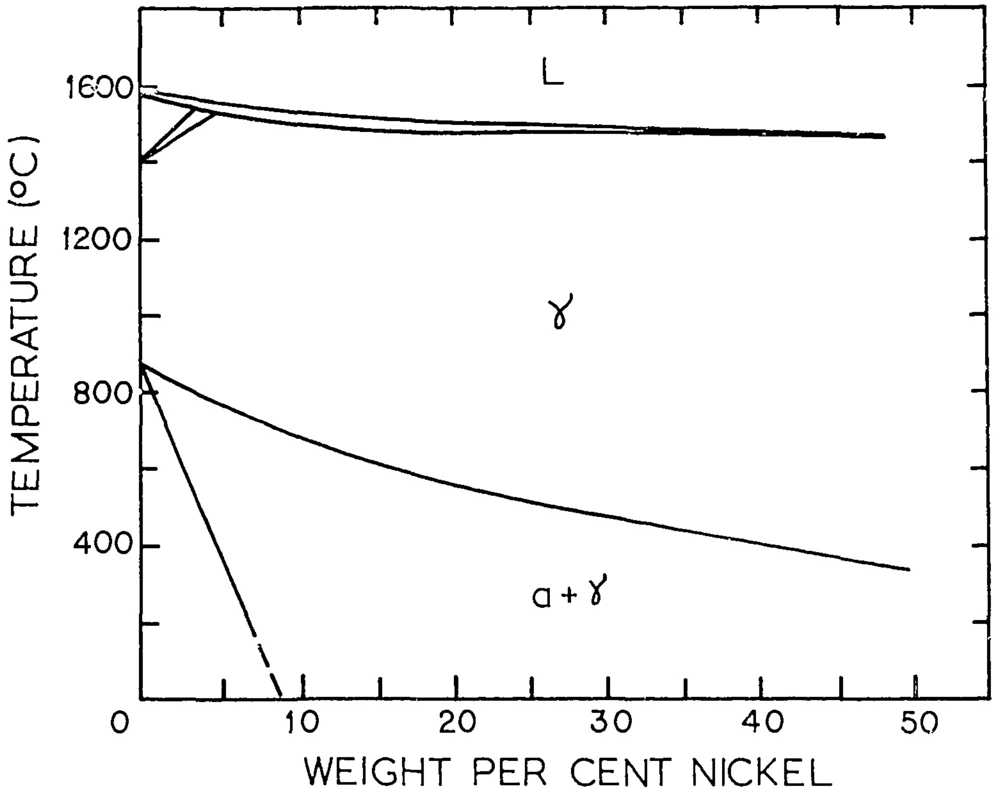


FIGURE 1a. IRON-RICH PORTION OF Fe-Ni EQUILIBRIUM DIAGRAM
(After Owen and Humphrey 1949)

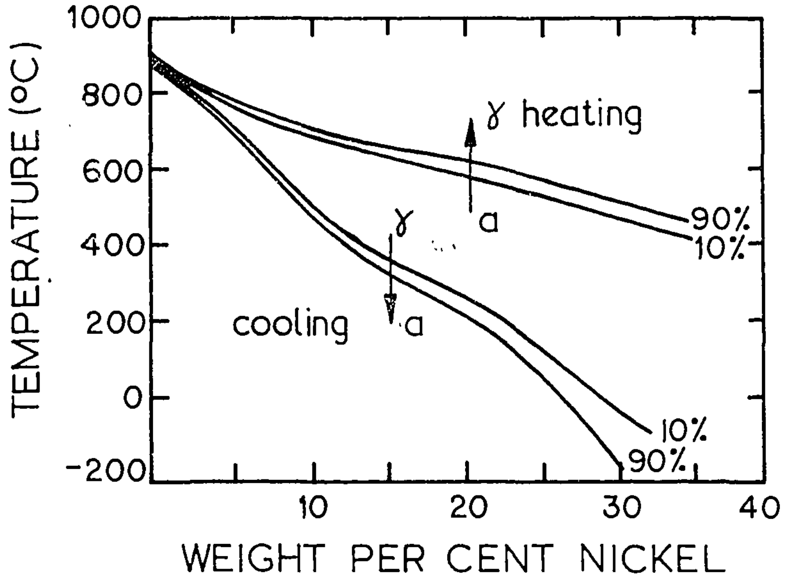
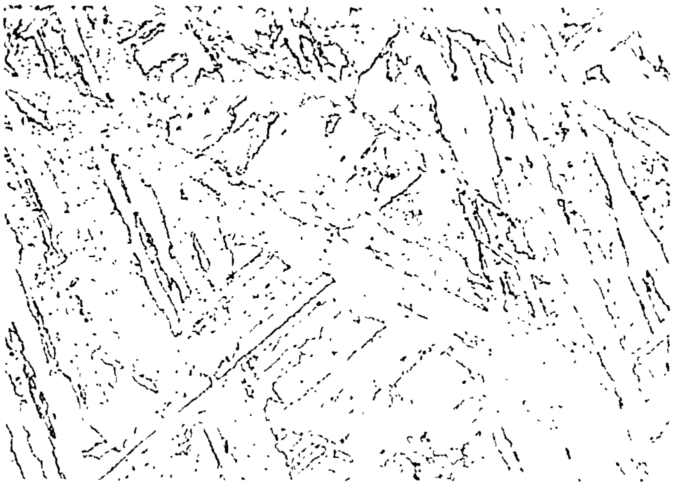


FIGURE 1b. METASTABLE IRON-NICKEL DIAGRAM
(After Jones and Liu 1949)



×500

FIGURE 2a. INHOMOGENEOUS DENDRITE STRUCTURE.
ARC-MELTED TWICE

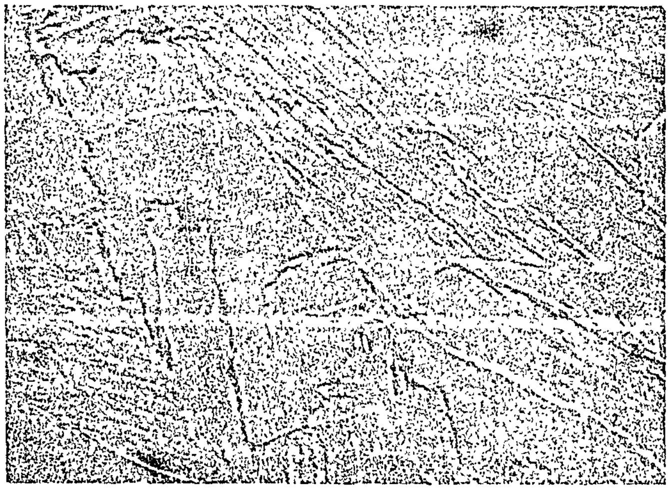


FIGURE 2b. STRUCTURE HOMOGENISED AT 1150°C.
ARC-MELTED TWICE

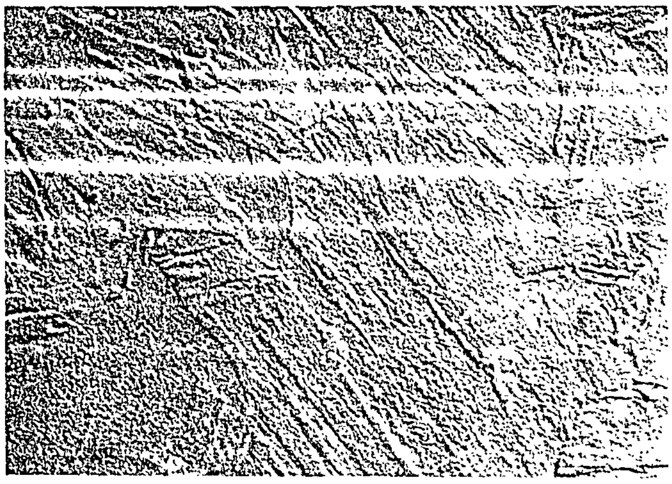


FIGURE 3a. KOBE MAR 350 AS-RECEIVED MATERIAL. HOT-WORKED ABOVE 900°C

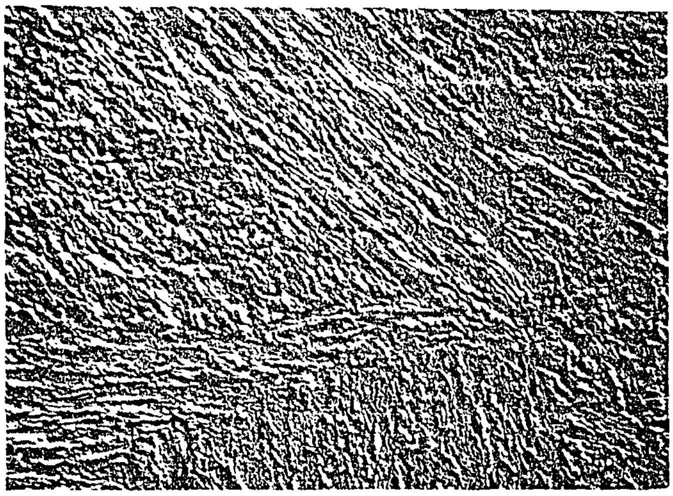


FIGURE 3b. SANDVIK MAR 350 COLD-ROLLED MATERIAL

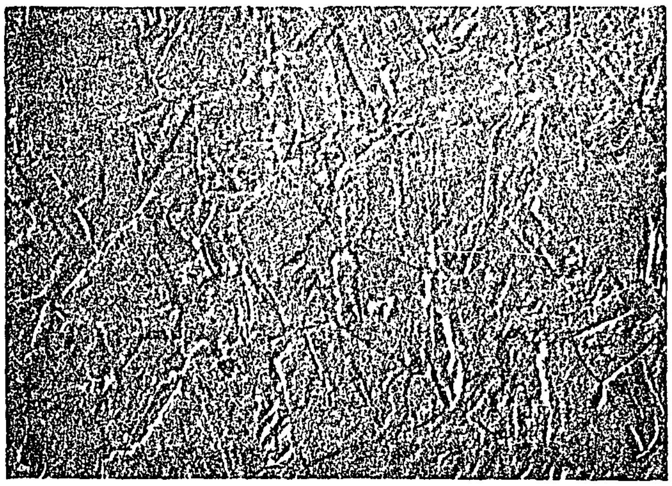
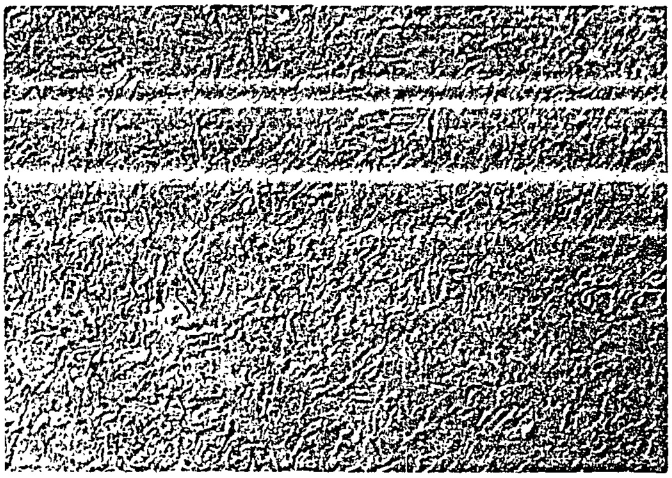


FIGURE 3c. SANDVIK MAR 350 COLD-ROLLED MATERIAL FOLLOWED BY SOLUTION TREATMENT AT 900°C FOR 60 MINUTES

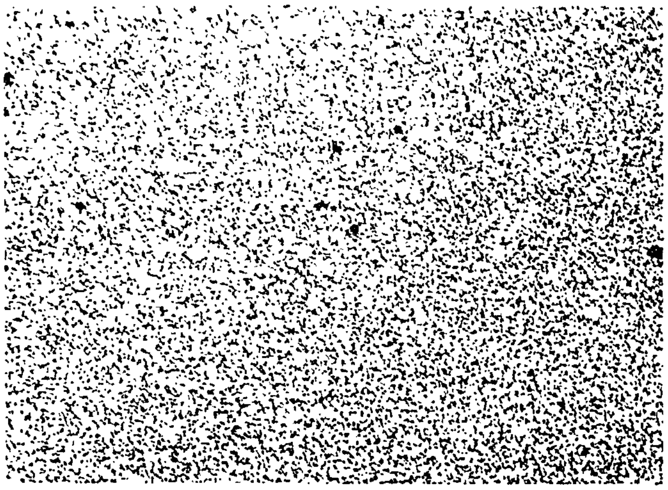


×500

FIGURE 4a. SANDVIK MAR 350. SOLUTION-TREATED FOR 60 MINUTES AT 815°C FOLLOWED BY AGEING AT 500°C FOR 180 MINUTES

×500

FIGURE 4b. SANDVIK MAR 400. SOLUTION-TREATED AT 1000°C FOR 60 MINUTES FOLLOWED BY AGEING AT 500°C FOR 180 MINUTES



×500

FIGURE 4c. SANDVIK MAR 400. SOLUTION-TREATED AT 1000°C FOR 60 MINUTES FOLLOWED BY AGEING AT 865°C FOR 120 MINUTES

Printed by the AAEC Research Establishment