DEPARTMENT OF
ENERGY, MINES AND RESOURCES
MINES BRANCH
OTTAWA

DYNAMIC RECOVERY OF ALUMINUM DURING HOT ROLLING

J. P. IMMARIGEON AND H. J. McQUEEN

PHYSICAL METALLURGY DIVISION

Reprinted from the Canadian Metallurgical Quarterly,
Vol. 8 No. 1, January - March 1969

Reprint Series RS 89 Price 25 cents

Available by mail from the Queen's Printer, Ottawa,
and at the following Canadian Government bookshops:

HALIFAX
1735 Barrington Street

MONTREAL
Æterna-Vie Building, 1182 St. Catherine St. West

OTTAWA
Daly Building, Corner Mackenzie and Rideau

TORONTO
221 Yonge Street

WINNIPEG
Mall Center Bldg., 499 Portage Avenue

VANCOUVER
657 Granville Street

or through your bookseller

Price 25 cents Catalogue No. M38-8/89

Price subject to change without notice

The Queen's Printer
Ottawa, Canada
1969

J.P. Immarigeon and H.J. McQueen

Dynamic recovery of aluminum during hot rolling

Abstract. Superpurity aluminum was rolled at 235 and 315°C to 90 per cent reduction in a single pass. The changes in microstructure arising from the deformation were examined by polarized-light and transmission-electron microscopy. The original grains were elongated in the direction of rolling and contained an equiaxed polygonized substructure. The subgrains were larger and more perfect at the higher rolling temperature. The extent of dynamic recovery is compared with that of commercial-purity aluminum and with that of other face-centred cubic metals.

Résumé. Un taux de réduction de 90% par laminage en une seule opération a été effectué sur de l'aluminium à 99.99% de pureté à 235 et à 315°C. Les modifications de la structure dues à cette déformation ont été éxaminées en lumière polarisée et par microscopie électronique en transmission. Les grains originaux sont allongés dans le sens de la déformation et présentent une sous-structure polygonalisée équiaxiale. Des sous-grains plus réguliers et plus gros ont été obtenus à la température la plus élevée. L'importance de la restauration dynamique est comparée avec celle de l'aluminium de pureté commerciale et avec celle d'autres métaux cubiques à faces centrées.

High-temperature forming is used extensively in industry as it allows a large reduction at a relatively low stress and without intermediate annealing. Theories (1-6) of the mechanism of hot working have been formulated, which were based on microstructural investigations and on the interdependence of strain, strain-rate, stress and temperature. Sellars and Tegart (1) found that dynamic recovery plays an important role in hot deformation and appears to be the rate-controlling process in aluminum; however, recrystallization may be the rate-controlling process in metals of low stacking-fault energy, such as copper and nickel. Stüwe (2) proposed a mechanism of high-temperature deformation based on the climb of edge dislocations into subgrain boundaries where they were annihilated. Work by Jonas, McQueen and Wong (3-6) clarified the relationship between the deformation parameters and the substructures in the extrusion of aluminum.

In the present study the aluminum was rolled to 90 per cent reduction in a single pass at two different temperatures. The resulting structures were examined by optical and electron microscopy. The results are discussed in comparison with those obtained in hot rolling (7-9) and in extrusion (3-7).

Experimental techniques

Specimen preparation and deformation. The metal employed was superpurity aluminum (99.99% Al). The specimens were machined as shown in Figure 1, to assist entry into the rolls. The specimens were annealed for at least 12 hours at 0.95 Tm (Tm: melting temperature, °K), which resulted in a grain size of 1 to 4 mm. They were heated to 0.95 Tm prior to rolling, for roughly half an hour.

The specimens (Figure 1) were rolled in a single pass, to reductions of 86 to 89 per cent, thus undergoing true strains between 2.0 and 2.3. Throughout the experiments, the roll speed (25 rpm) and diameter (3 inches) were kept constant, giving an average strain rate of 20 sec^{-1}. Two roll temperatures were used: 25 and 250°C; the latter was obtained by electric heaters on the outer side of each roll (Figure 2). The specimen was pulled from the tube furnace into the rolls by a wire embedded in the tapered side. The temperature was measured by a thermocouple which was embedded in the thicker part and which was fed in from the other end of the furnace. The narrow section of the specimen was immediately engaged by the rolls and the remainder was drawn in. The emerging sheet was supported by the part of the sample still in the rolls, until the whole

*The essay from which this paper is an extract was awarded the 1968 prize for the student essay in physical metallurgy by the Canadian Institute of Mining and Metallurgy. The micrographs of Figure 5 were recognized as the best student entry in the 1967 metallography contest of the American Society for Metals.

J.P. Immarigeon, student assistant, Physical Metallurgy Division, Department of Energy, Mines and Resources, Ottawa, Canada. Presently graduate student, Department of Metallurgical Engineering, McGill University, Montreal.

H.J. McQueen, research scientist, Physical Metallurgy Division, Department of Energy, Mines and Resources, Ottawa. Presently Associate Professor of Materials Science, Mechanical Engineering, Sir George Williams University, Montreal.

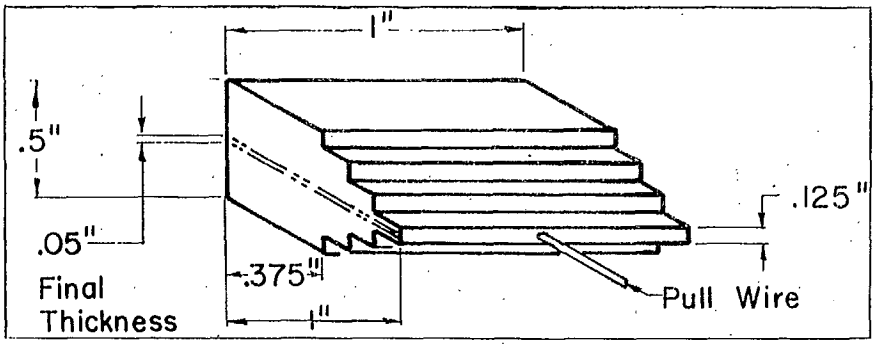


Figure 1. Hot-rolling specimen.

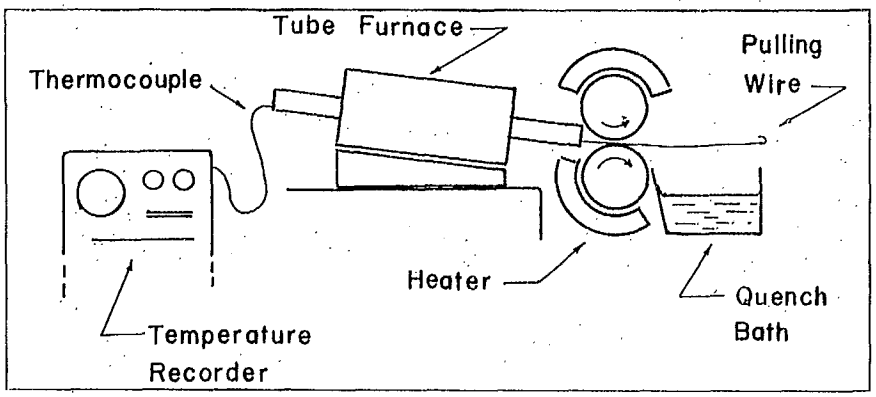


Figure 2. Experimental setup for hot rolling.

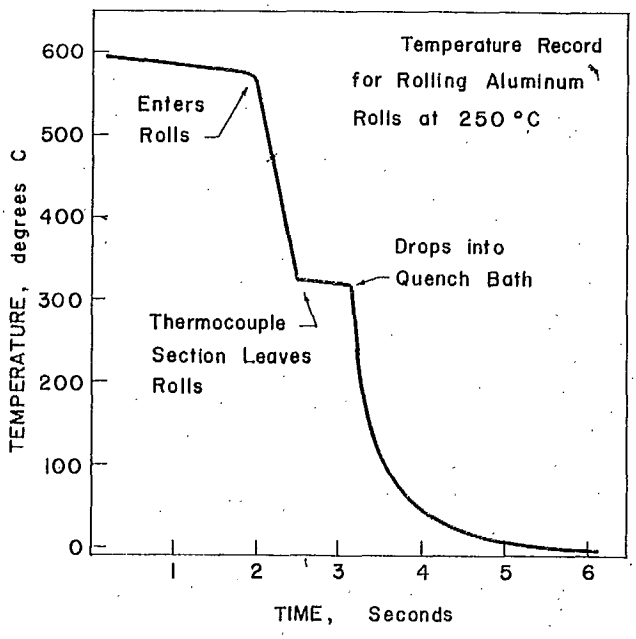


Figure 3. Temperature variation of aluminum during rolling on rolls at 250°C to result in a finishing temperature of 0.65 Tm (°K).

specimen emerged. It then dropped into a bath of iced brine (−10°C), the quenching occurring in less than 3 seconds. A major difficulty was encountered in rolling the aluminum: samples stuck to the rolls probably because of welding after the breakup of the oxide layer. This occurred less readily when the rolls were at 250°C.

The temperature variation of the specimen during rolling (Figure 3) was recorded on a high-speed photographic galvanometer. Figure 3 shows that the time lapse between the emergence from the rolls and the quench was less than 0.5 second. However, since the cooling in air was slight and since the sheet did not immediately fall into the quench bath, the time at finishing temperature varied along the length of the specimen. Nevertheless, the rear part of the sample can be considered to have been quenched in less than 0.5 second. The average specimen temperatures during rolling were 0.70 and 0.75 Tm. The finishing temperatures of 0.55 and 0.65 Tm are quoted since they represent the minimum working temperatures; these are still within the normal hot-working range. Furthermore, the finishing temperature determines the structural alterations that occur between cessation of deformation and quenching. It is interesting to note on Figure 3 the severe quench that occurred during rolling such a thin sample to a high reduction. When the rolls were used cold (25°C) the finishing temperature was 235°C, whereas when the rolls were heated up to 250°C the finishing temperature was 315°C, an increase of only 80°C. A higher finishing temperature was not feasible, for fear of softening the rolls.

Specimens from the rolled sheets were annealed for 1 minute and 5 minutes at their respective temperatures to observe the progress of recrystallization.

Transverse sections parallel to the rolling direction were examined by optical metallography and samples from the plane of rolling were examined in the electron microscope. Samples were taken from an area cooled in less than 1 second and others from an area cooled in 2 to 4 seconds.

Optical metallography. Several techniques are described in the literature (10, 11); however, a combination of these was found to give the best results. The rolled-sheet sections, mounted on their edges, were polished on carbide paper and finally on cloth with 9 μm diamond. The electrolytic polish was performed at 18 to 20 volts and 3 to 4 amps per square inch for 2 to 2.5 minutes in a solution of 20 per cent perchloric acid and 80 per cent ethyl alcohol by volume. The electrolyte was maintained at about 12°C because it becomes explosive above 35°C. The stainless steel cathode should be about 3/8 inch from the specimen. To provide electrical contact with the specimen, which did not adversely affect the polishing conditions, a screw was inserted through the nonconducting mount and was preserved from contact with the electrolyte. When fresh electrolyte was used, a piece of pure aluminum was polished for at least 5 minutes before using the bath for a rolled section.

The technique to reveal the microstructure utilizes the birefringent properties of an electrolytically produced anodic film and requires polarized light for examination (12). The anodizing solution was 2 per cent hydrofluoric acid (48% HF), 49 per cent ethyl alcohol and 49 per cent distilled water by volume. An electrolysis of 30 seconds at 40 to 50 volts, with a current density of .4 to .5 amp per square inch (12) was found sufficient to give a good anodic film, free of pitting. The sections were observed under polarized illumination on a Bausch and Lomb metallograph and photographed with the rolling direction horizontal.

Electron microscopy. Samples cut from the rolled sheet, 1.3 mm thick, were wet ground to a thickness of roughly 0.7 mm. During chemical thinning to about 0.3 mm in a solution of 25 per cent sulphuric acid, 70 per cent phosphoric acid and 5 per cent nitric acid by volume at 85°C, the sample orientation was repeatedly altered to produce a uniform thickness. Discs for insertion in the electron microscope were cut from the foil by a rotating jet of 10 vol. per cent nitric acid in water at a potential of 100 volts. A small bridge was preserved between the foil and the disc for electrical contact during subsequent polishing. A larger stationary jet and 80 volts were used to prepare a relatively flat-bottomed dish with a thickness of 200 μm in the central part of the disc. To preserve the bridge between the disc and the foil, to protect the remaining foil surface, and to prevent contact between the tweezers and electrolyte which resulted in high current density, gas evolution and pitting, the tweezers, foil and bridge were varnished with 'Microstop' with only the disc left unprotected. Polishing in a 20 per cent perchloric acid, 80 per cent ethanol solution by volume, cooled to about 10°C, at a potential of 14 to 19 volts with a stainless steel cathode (13-15), was continued until perforation was observed through a telescope. The discs were removed from the foil by cutting the bridge and were examined in transmission, in a Siemens electron microscope equipped with a Valdré double tilting stage.

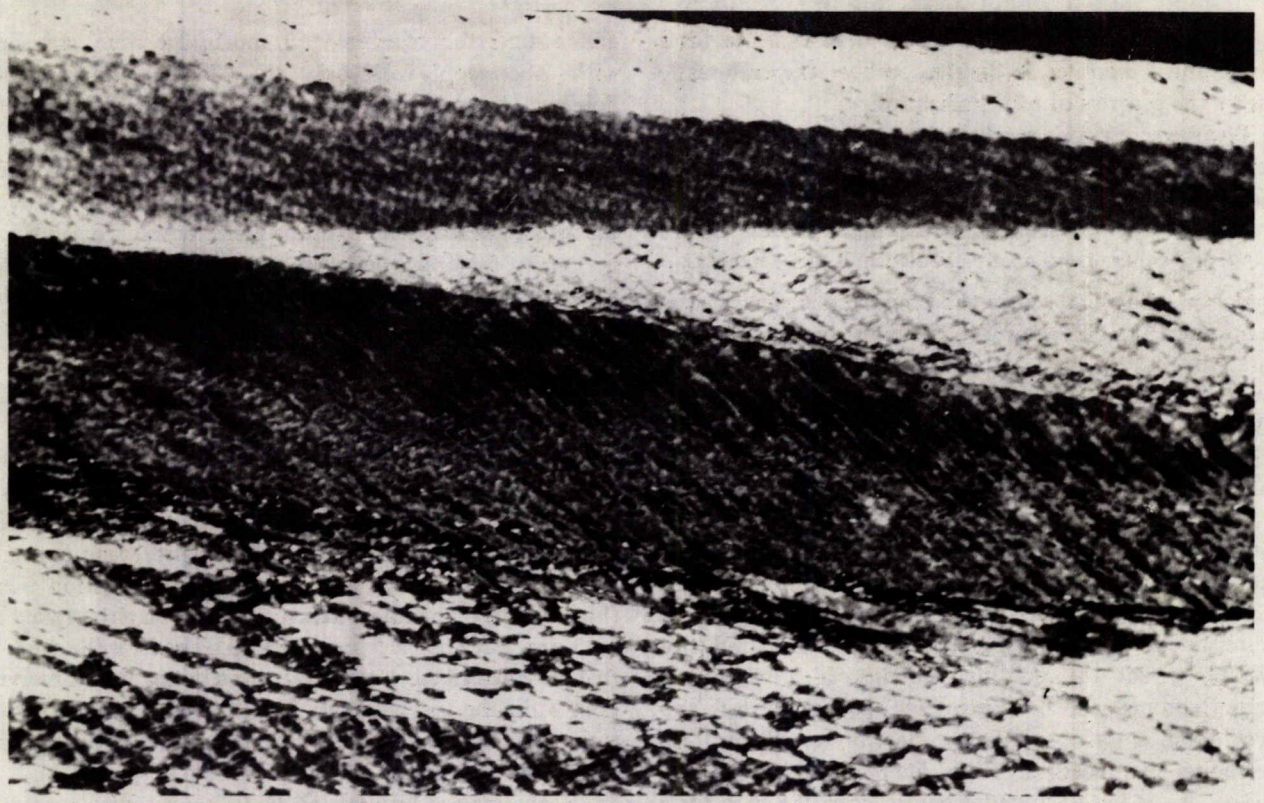
Experimental results

Optical metallography. The microstructure resulting from rolling at 0.55 Tm and quenching in less than a second is a worked structure. Figure 4(a) shows well the distorted elongated grains. The use of polarized light reveals not only the fibrous grains but also the substructure within them (Figure 4(b)). The microstructure of the material rolled at 0.55 Tm, but cooled in more than 2 seconds, is the same as that just described. No recrystallized grains were observed even when the specimen was cooled in air. The microstructure of the sample rolled at 0.65 Tm is basically the same as the one resulting from rolling at 0.55 Tm (Figure 4(c)).

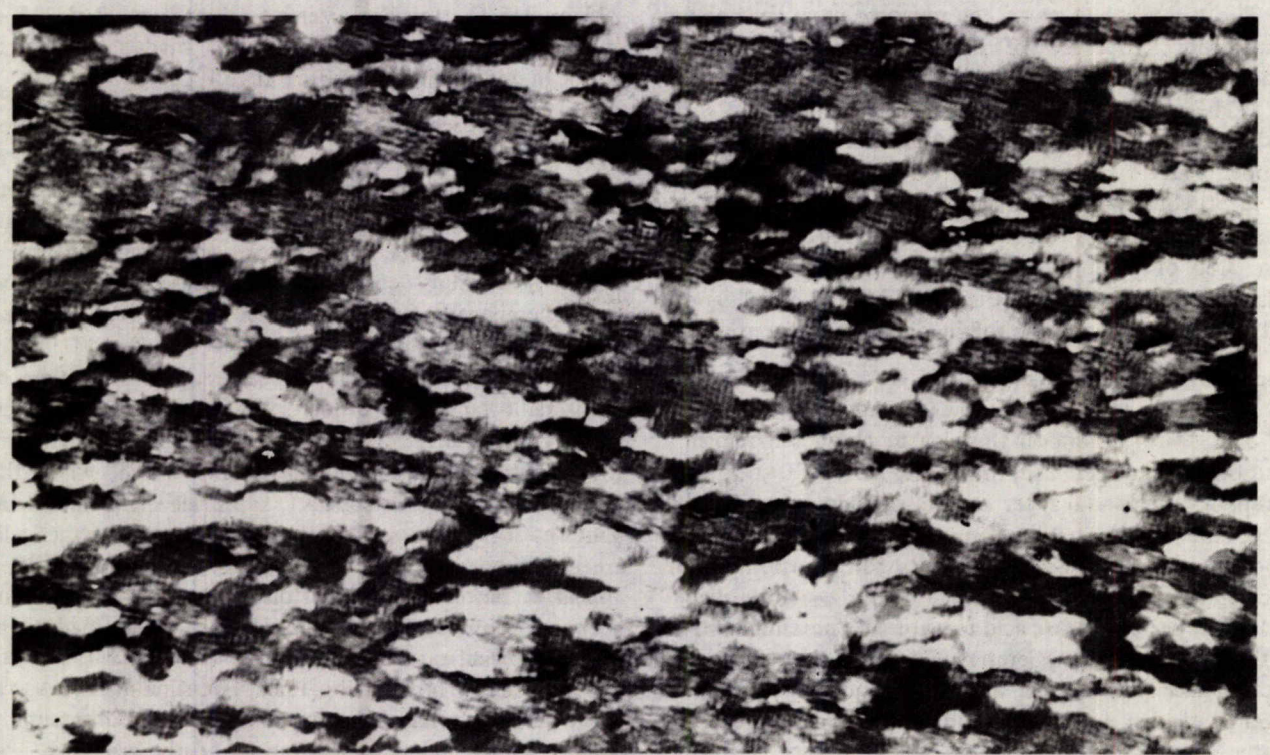
Reheating for 1 minute at the finishing temperature did not initiate recrystallization in specimens finished at either 235 or 315°C. However after a 5-minute anneal at 315°C about 10 per cent recrystallization had taken place, although none had yet appeared at 235°C.

Electron microscopy. The substructures observed by transmission electron microscopy for each rolling temperature are shown in Figure 5. Areas of low dislocation density are outlined by boundaries of high dislocation density. As the temperature increases, the subgrains are larger and have a lower dislocation density. In both cases the subgrains appear equiaxed. No recrystallized grains were observed whether quenching was immediate or delayed. At 0.55 Tm the sub-boundaries usually appear ragged, consisting of fairly tight tangles of dislocations; occasionally neat networks were observed (Figure 6). At the higher temperature, they consist of dislocation networks which, when the dislocations are not in contrast, appear as sharp lines or as bands of fringes like high-angle boundaries. When the walls are at the correct angle of tilt to the electron beam they appear as simple networks of dislocations. Misorientations between neighbouring subgrains were determined from selected-area diffraction made with apertures of 30 or 100μm exposing one of several subgrains. Rotation about an axis perpendicular to the rolling plane was measured by simply comparing the relative orientation of the patterns. The rotation was usually less than 1° and never exceeded 4°.

In a few cases, while a foil was being examined, dislocations were seen gliding across subgrains and crossing sub-boundaries. This effect occurred only in cases where



(a)



(b)

(c)

Figure 4. Microstructures observed under polarized light of aluminum hot-rolled at 0.55 Tm (150X) (a), (1000X) (b), and at 0.65 Tm (1000X) (c).

the anticontamination device warmed up and became ineffective. These displacements are easily observed as each dislocation leaves a trace of its path, which disappears after a few minutes. In the photographs of Figure 7 the dislocations did not move in a straight line along a definite slip plane but turned away, by repeated cross slip, from the top sub-boundary, which repelled them. The passage of dislocations across sub-boundaries without noticeable change in direction is further evidence that neighbouring subgrains have almost the same orientation.

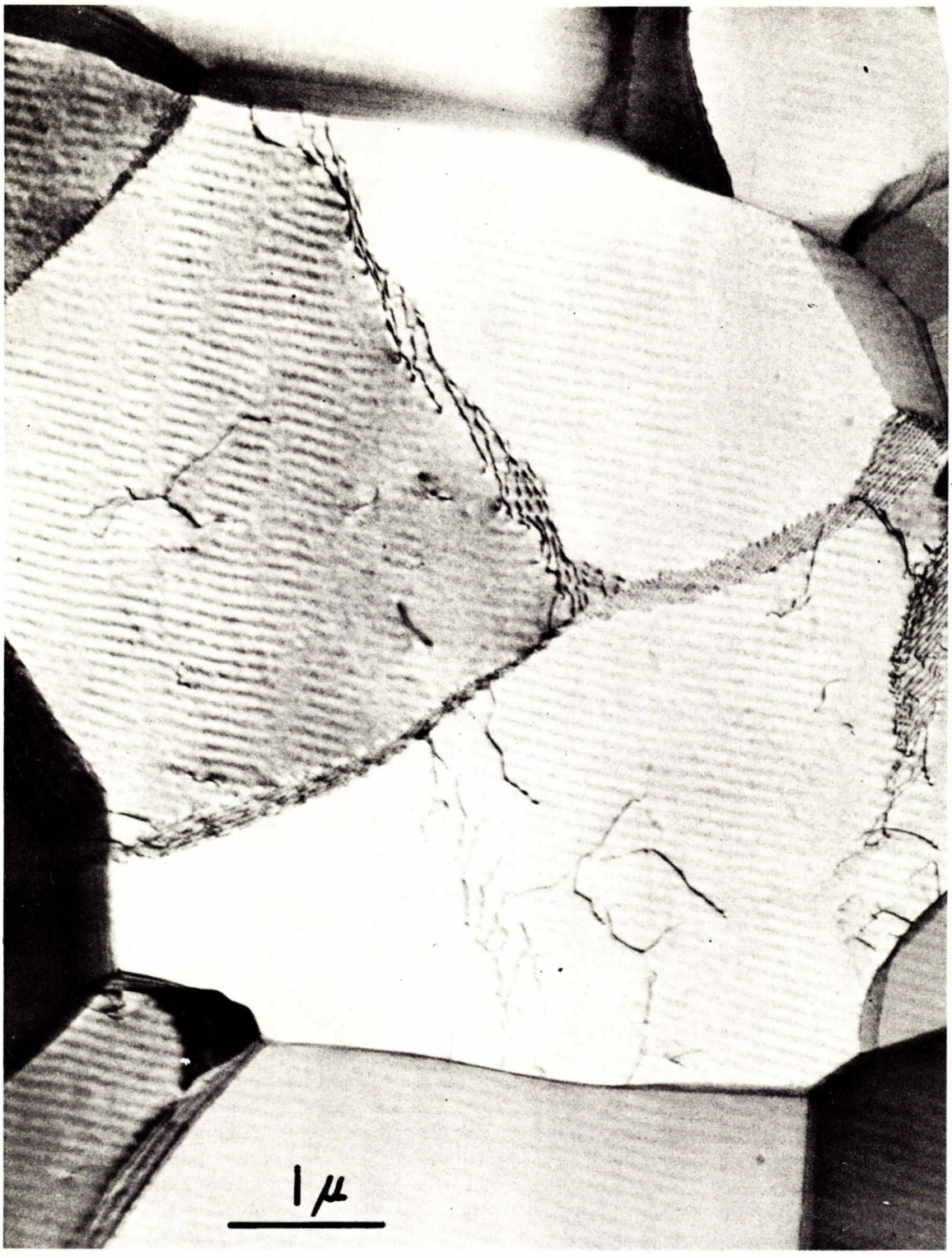
Discussion

During rolling, fibrous worked grains containing a substructure were produced. In general, the microstructures are substantially similar to those observed after extrusion (3-7) and hot rolling (7, 8), even though there was considerable difference in the strain-rate and the strain. Although the polarized light reveals a substructure, previous research (3, 4, 17) showed that it does not give an unambiguous indication of size or relative misorientation of subgrains. The 'optical subgrains', which are rendered visible in the polarized-light technique through variations in the orientation of an anodized layer, cover many of the true subgrains which are revealed by transmission-electron microscopy. The anodized layer apparently nucleates on only a fraction of the subgrains then grows across sub-boundaries until it impinges upon other patches. The observation of the substructure is useful in facilitating recognition of recrystallized grains which contain no substructure.

Recrystallization did not occur in aluminum after hot rolling even when held 1 minute at finishing temperature; it appeared after a 5-minute anneal only at 0.65 Tm. Such resistance to recrystallization has been observed by other researchers (3, 7, 8, 18); the worked microstructure produced by hot extrusion at 400°C did not recrystallize on subsequent annealing at 350°C for 1 hour (3). The presence of the well formed subgrains in hot-rolled aluminum shows that polygonization took place during the process. This high recovery seems to confer a resistance to recrystallization which is much greater than that of fcc metals of lower stacking-fault energy, such as nickel, copper and brass (9).

The results of the investigations carried out on the electron microscope are in complete agreement with those of previous researchers. Equiaxed subgrains have been observed in extruded aluminum (3) and in aluminum deformed by hot torsion (19). For similar conditions of deformation, the subgrains in superpurity aluminum are much larger and more perfect than in commercial purity (3). The increase of the subgrain size following an increase in deformation temperature has been reported by other researchers for hot rolling (7), for hot torsion (19) and for

(a)



(b)

Figure 5. Electron micrographs of aluminum rolled at 0.55 Tm (a) and at 0.65 Tm (b).

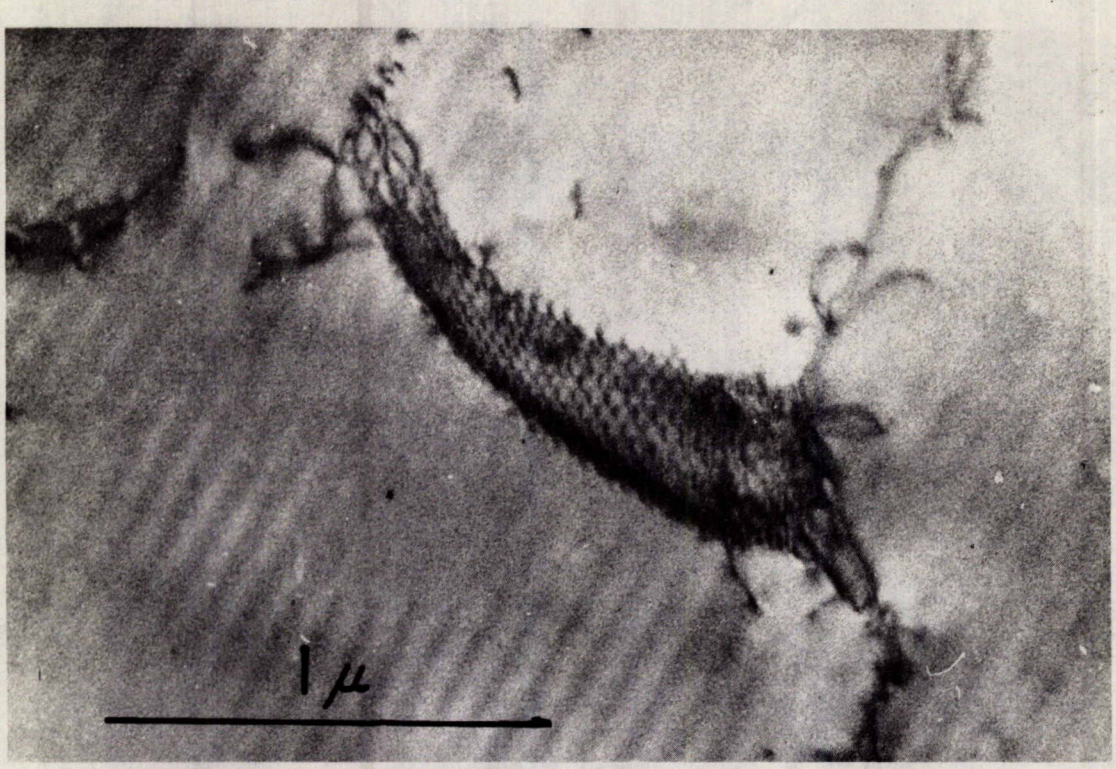


Figure 6. Hexagonal dislocation array in aluminum rolled at 0.55 Tm.

hot extrusion (3). Wong, *et al.* (3) also reported an increase in internal perfection of the subgrains with rising extrusion temperature. McQueen (9) observed a similar trend in other fcc metals; however, the polygonization in them was much less because of the lower stacking-fault energy. The misorientation between neighbouring subgrains compares well with results obtained for extrusion (3). The formation of subgrains whose size and internal perfection increase with rising rolling temperature leads to lower forming forces, since the strength is inversely proportional to the subgrain size (5).

In light of the understanding of earlier researchers, Stüwe (1), Jonas, *et al.* (4) and McQueen, *et al.* (5, 9, 20), the presence of an undistorted, equiaxed, polygonized cell structure with low misorientations shows that dynamic recovery prevails during rolling. Dynamic recovery is softening concurrent with deformation, particularly at high stresses and strains. At a temperature above one half of the absolute melting point, dynamic recovery causes the true stress-true strain curve to be roughly horizontal, after an initial hardening strain of approximately unity (1, 2). Since the specimens were rolled to a strain greater than 2, the deformation passed into the above region of negligible strain hardening. The initial deformation causes the dislocations to climb, glide and cross-glide to form tangles and networks resulting in hardening. Furthermore, the strain enhances both the climb rate, by the production of vacancies, and the rate of annihilation, by the build-up in dislocation density. Ultimately a balance is achieved between hardening and recovery which maintains the dislocation density constant as subgrains of constant size and misorientation, and permits flow at a constant strain rate without change in flow stress. As the subgrains do not appear elongated, it was concluded that during the steady-state deformation the sub-boundaries continuously break up and reform. Dislocations bow out from sources in the walls or interior of subgrains and move by glide, cross glide and climb across many subgrains. Breaking-up and reforming of sub-boundaries (repolygonization) go on as dislocations cut through boundaries or come to rest in those in which they combine or are annihilated.

As the rolling temperature was increased from 0.55 to 0.65 Tm, the size of the subgrains was increased and the sub-boundaries sharpened. This occurred with the increased ability of the dislocations to rearrange into simpler, neater networks by climb, crossglide, combination and annihilation. The lower dislocation density at the higher rolling temperature results in a lower flow stress.

Conclusions

The structural examination of hot-rolled aluminum lead to the following general conclusions:

1. Aluminum rolled at 0.55 and 0.65 Tm does not recrystallize even when held at finishing temperature for 1

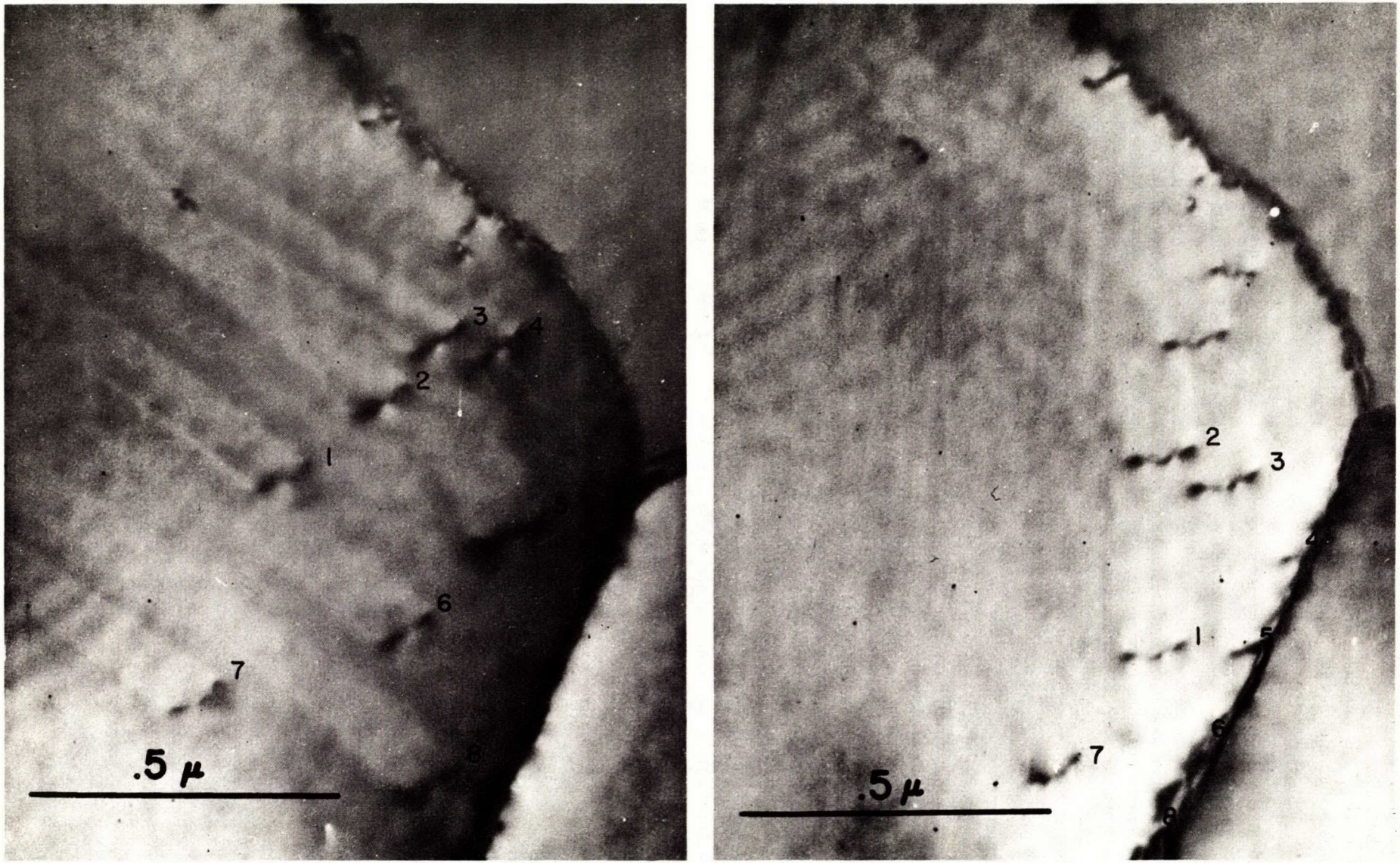


Figure 7. Successive positions of a group of dislocations (identified by numbers) is shown in micrographs a and b. The curved path is the result of repeated fine cross slip. There is an interval of about one minute between (a) and (b).

minute. This resistance to recrystallization is due to high recovery during hot rolling.

2. Optical metallography with polarized light reveals a fibrous worked structure and indicates the presence of a substructure. However, it does not give reliable measurements of subgrain size or misorientation.

3. Electron microscopy reveals equiaxed subgrains of which the size and the neatness of the boundaries increase with increasing temperature.

4. Since the substructures are similar to those observed after hot extrusion and hot torsion, dynamic recovery must also be the mechanism of hot rolling of aluminum.

5. When compared to other face-centred cubic metals rolled under similar conditions, aluminum shows a higher recovery, which is attributed to its high stacking-fault energy.

Acknowledgments

Grateful acknowledgments must be made to Dr. E. Laufer for discussion and advice on electron microscopy; to B. Renaud for initiation to electron microscopy; to E.F. Connors for advice on metallographic preparation; to R.I. Hamilton for temperature measurements; and to all members of the Metal Physics Section, Department of Energy, Mines and Resources, Ottawa, for their constant help.

References

(1) Sellars, C.M., and W.J. McG. Tegart. *Acta. Met.* 14: 1136, 1967.
(2) Stüwe, H.P. *Acta. Met.* 13: 1337, 1965.
(3) Wong, W.A., H.J. McQueen, and J.J. Jonas. *J. Inst. Met.* 95: 129, 1967.
(4) Jonas, J.J., H.J. McQueen, and W.A. Wong. *Deformation Under Hot Working Conditions*, publication 108, Iron and Steel Inst., London, 1968, p. 49.
(5) McQueen, H.J., W.A. Wong, and J.J. Jonas. *Can. J. Phys.* 45: 1225, 1967.
(6) McQueen, H.J., W.A. Wong, and J.J. Jonas. *Acta. Met.* 15: 586, 1967.
(7) Leguet, R., D. Whitwham, and J. Hérenguel. *Mém. Scient. Rev. Mét.* 59: 649, 1962.
(8) McLean, D., and A.E.L. Tate. *Revue Métal.* 48: 765, 1951.
(9) McQueen, H.J. *Proc. Intl. Conf. on Strength of Metals and Alloys. Trans. Japan Inst. Met.* 9, supplement: 171. 1968.
(10) Tegart, W.J. McG. *Electrolytic and Chemical Polishing of Metals in Research and Industry.* London, Pergamon, 1959. p. 29.
(11) Hamilton, R.I., and E.F. Connors. *The Preparation of 'As Polished' Metallographic Finishes in Nonferrous Metals.* Inf. Circular IC176, Phys. Met. Div., Dept. Energy, Mines and Resources, Ottawa, 1965.
(12) Barker, J.L. *Trans. ASM.* 42: 347, 1950.
(13) Boswell, F.W.C., and E. Smith. *Symp. on Advances in Electron Metallography,* STP No. 245, ASTM, Philadelphia, 1958, p. 21.
(14) Hirsch, P.B., A. Howie, R.B. Nicholson, D.W. Pashley and M.J. Whelan. *Electron Microscopy of Thin Crystals.* London, Butterworths, 1965.
(15) Thomas, G. *Transmission Electron Microscopy of Metals.* New York, John Wiley, 1965.
(16) Wong, W.A. Master's thesis, McGill University, 1964.
(17) Jonas, J.J., H.J. McQueen, and D.W. Demianczuk. *Deformation Under Hot Working Conditions*, publication 108, Iron and Steel Inst., London, 1968, p. 97.
(18) Schey, J. Acta Tech. Acad. Sc. Hung. 16: 131, 1957.
(19) Ormerod, H., and W.J. McG. Tegart. J. *Inst. Met.* 92: 297, 1963-1964.
(20) McQueen, H.J. *J. Met.* 20: (4):31. 1968.