TABLE I Lattice constant and crystallite size values of CaO microcrystals

Temperature (° C)	Atmosphere	Lattice* constant a (Å)	Change in lattice constant Δa (Å)	Crystallite size (Å)	N
27	vacuum	4.8103	−0.0002†	95	41
325	vacuum	4.8025	−0.0080†	142	60
562	vacuum	4.7991	−0.0114	158	66
795	vacuum	4.7999	−0.0106	175	73
874	vacuum	4.7981	−0.0124	179	75
874	vacuum	4.7981	−0.0124	183	76
874	vacuum	4.8027	−0.0078	245	102
27	vacuum	4.8093	−0.0012	246	103
27	moist air	4.8127	+0.0022	246	103

* Lattice constant corrected to 27° C.
† Minimum lattice parameter values not reached due to incomplete removal of hydroxyl ions (see text).

present at temperatures up to at least 600° C. There is some evidence to suggest that the size of the original hydroxide platelets influences the initial size of the oxide microcrystals [11], hence smaller hydroxide crystallites are necessitated. However, a more fruitful approach may rest on decomposition under ultra-high vacuum at low temperatures.

Acknowledgement

The authors are grateful for the bursary awarded to one of them (I.F.G.) by the UKAEA.

References

1. I. F. GUILLIATT and N. H. BRETT, *Trans. Faraday Soc.* **65** (1969) 3328.
2. P. J. ANDERSON and A. SCHOLZ, *ibid* **64** (1968) 2973.
3. M. M. NICOLSON, *Proc. Roy. Soc.* **228** A (1955) 490.
4. A. CIMINO, P. PORTA, and M. VALIGI, *J. Amer. Ceram. Soc.* **49** (1966) 152.
5. R. C. GARVIE, *Mat. Res. Bull.* **1** (1966) 161.
6. E. R. PIKE and A. J. C. WILSON, *Brit. J. Appl. Phys.* **57** (1959) 10.
7. J. W. HARRISON, AERE Report 4918 (1965).
8. P. T. B. SCHAFFER, "High Temperature Materials, Index" (Plenum Press, New York, 1964), p. 327.
9. I. F. GUILLIATT and N. H. BRETT, *Phil. Mag.* **21** (1970) 671.
10. P. J. ANDERSON, R. F. HORLOCK, and R. G. AVERY, *Proc. Brit. Ceram. Soc.* **3** (1965) 33.
11. I. F. GUILLIATT, unpublished work.

Received 5 March
and accepted 17 April 1970.

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*Subgrain Growth on Annealing of Thin Foils of Cold-rolled Aluminium**

The mechanism of subgrain growth on annealing of cold-rolled metals has been studied by several investigators after the invention of transmission electron microscopy. Reviews are found in [1] and [2]. Li [3] has proposed that subgrains grow by coalescence. The theory is supported by experimental observations made by Hu [4, 5] and Koo and Sell [6] in 3% silicon-iron and Weissmann [7] in aluminium. Walter and Koch [8, 9] have also studied 3% silicon-iron. They are of the opinion that subgrains grow by subboundary migration.

The author's intention was to investigate the problem of subgrain coalescence further. Thin foils of cold-rolled aluminium were observed

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* The experimental work was done at The Laboratory of Metallurgy, The Technical University of Denmark, Lyngby, Denmark. The author is now at The Technical University of Denmark, DIA-M, Lyngby, Denmark.

during heating by the beam of an electron microscope. A moderate and a strong degree of deformation, 30 % and 98 %, were used.

The subgrain growth mechanism observable on heating of a thin foil of a cold-rolled metal [4, 7] is probably not identical with the mechanism in bulk material; the very small thickness of the foil causes a great loss of dislocations, approx. 50 % in aluminium [10], and the dislocation movement is probably impeded by the oxide film on the surface. On the heating of a thin foil by the electron beam, thermal stresses are introduced into the foil, which may also contribute to another growth mechanism in thin foils differing from that in bulk material. However, Hu [4] and Weissmann [7] have studied subgrain growth by annealing of thin foils, and their results were not inconsistent with results obtained by annealing in bulk. Therefore it seems

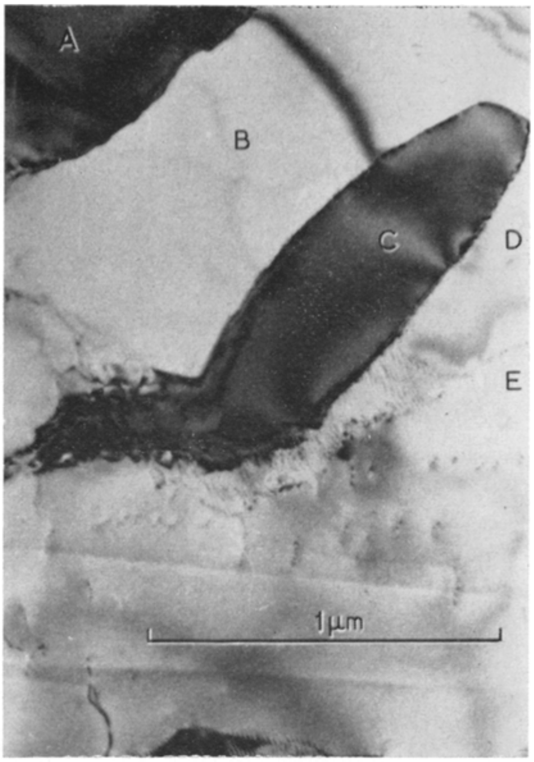

Figure 1

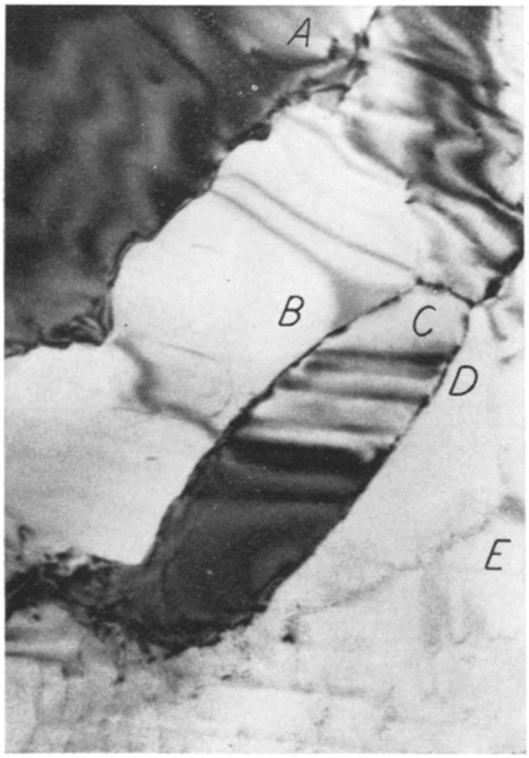

Figure 2

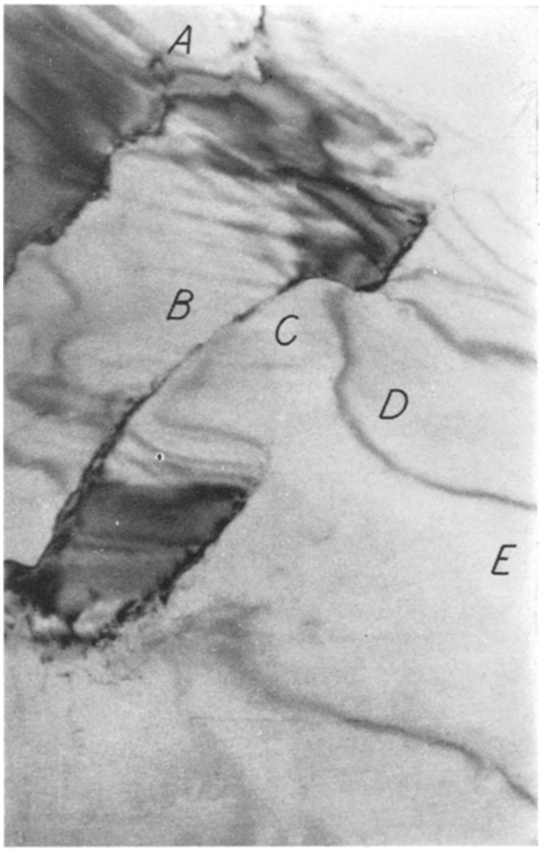

Figure 3

Figures 1-3 Aluminium deformed by 98%. Dislocation movements during heating of a thin foil by the electron beam.

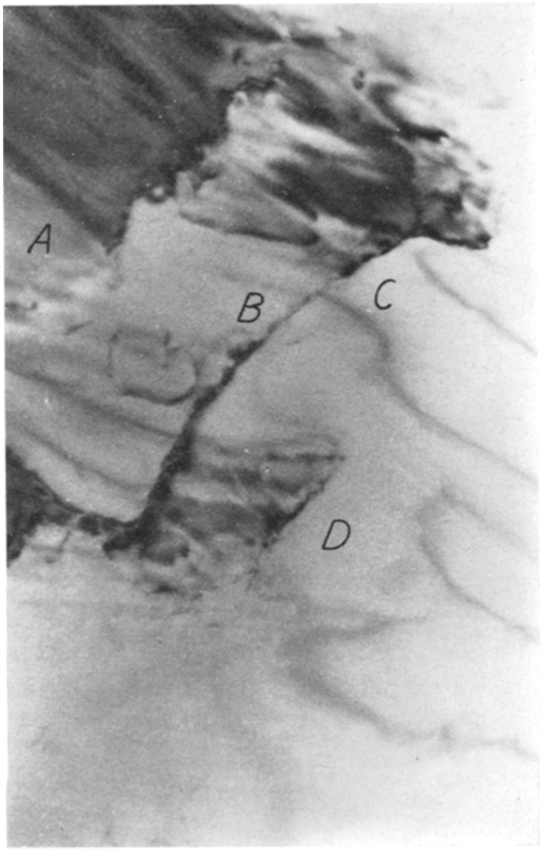

Figure 4

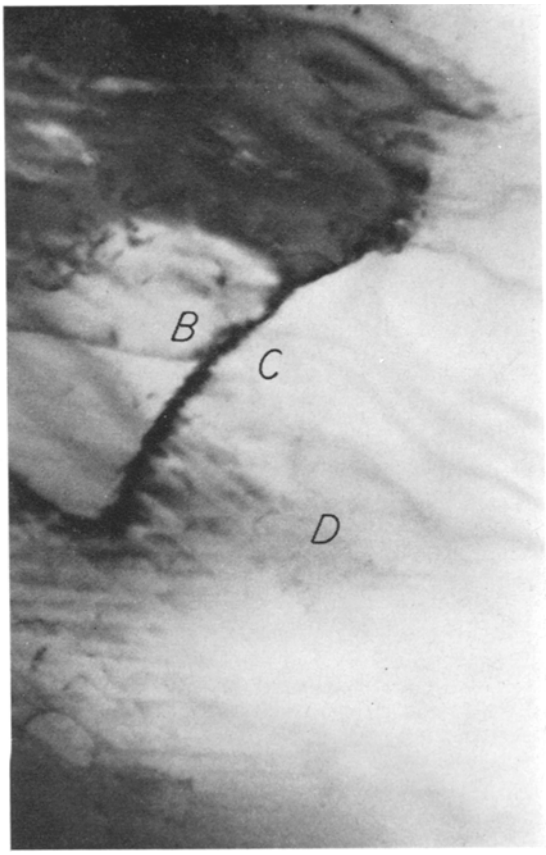

Figure 5

Figures 4-5 Dislocation movements during further heating of the thin foil in figs 1-3

reasonable to use annealing of thin foils to obtain indications of the character of the mechanism of subgrain growth.

Polycrystalline aluminium of 99.99% purity was used. Sheets deformed by 98% and 30% were produced by cold-rolling to 0.1 mm. Specimens for transmission electron microscopy in a Hitachi HU-11 A microscope were prepared.

Thin foils from cold-rolled specimens were heated in the electron microscope by the electron beam, which was concentrated as much as possible. The microscope was used without the specimen cooling stage to make the movements of dislocations easier. Many examples of the process shown in figs. 1-5 were seen; however, it was very difficult to produce photographs which showed the process as clearly as do figs. 1-5. Fig. 1 shows the original subgrain structure in 98% deformed material with the subgrains A, B, C, D, and E.

The heating was started, and dislocations were seen moving from the right to the left in subgrain C (fig. 2). The horizontal lines in subgrain C are slip traces. In fig. 3 the C-D boundary has become partly invisible. However, the curving of the bend extinction contour that runs over the sub-boundary shows that some dislocations are still left there. Dislocations have by now started to move in subgrains A and B. The upper, straight bend extinction contour over the C-D sub-boundary in fig. 4 shows that in that place the sub-boundary has disappeared. The lower bend extinction contour, still curved, proves that some dislocations are still left there. In fig. 5 grain C has practically the same crystallographic orientation as D because the two subgrains are not separated by dislocations. This was confirmed by tilting of the specimen in the electron microscope. In the last stage of the process dislocations moved on two slip systems.

In the process shown in figs. 1-5, the dislocations in sub-boundary C-D have moved mainly to sub-boundary B-C, which is indicated by the slip traces and the disappearance of sub-boundary C-D. This means that grain C has rotated to the same orientation as grain D. The observed phenomenon seems to be a sub-grain coalescence process giving experimental support to the coalescence model originally proposed by Li [3].

Acknowledgement

The author thanks Professor Dr E. Knuth-Winterfeldt, Professor Dr E. W. Langer and the other members of the staff at the Department of Metallurgy of the Danish Technical University for helpful discussions. The author equally acknowledges assistance from members of the staff at the Department of Metallurgy of the Research Establishment Risö.

References

1. L. HIMMEL (editor), "Recovery and Recrystallization of Metals", Proceedings of a Symposium, New York, 1962. (Interscience, New York, 1962) 389 pp.
2. "Recrystallization, Grain Growth and Textures", papers presented at a Seminar of the American Society for Metals, Metals Park, Ohio, 1965. (American Society for Metals, Metals Park, Ohio, 1966) 617 pp.
3. J. C. M. LI, *J. Appl. Phys.* **33** (1962) 2958.
4. H. HU, *Trans. AIME* **224** (1962) 75.
5. J. C. M. LI, *op. cit.* "Recrystallization, Grain Growth and Textures", pp. 45-97.
6. R. C. KOO and H. G. SELL, *op. cit.* "Recrystallization, Grain Growth and Textures", p. 97-98.
7. S. WEISSMANN, T. IMURA, and N. HOSOKAWA, *op. cit.* "Recovery and Recrystallization of Metals", pp. 241-267.
8. J. L. WALTER and E. F. KOCH, *Acta Met.* **10** (1962) 1059.
9. *Idem, ibid* **11** (1963) 923.
10. P. B. HIRSCH, in, "The Relation between the Structure and Mechanical Properties of Metals", Proceedings of a Conference, Teddington, Middlesex, 1963. (HMSO, London, 1963) pp. 40-82.

Received 13 April
and accepted 16 April 1970.

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