

Hirsch P B, Silcox J, Smallman R E & Westmacott K H. Dislocation loops in quenched aluminium. *Phil. Mag.* 3:897-908, 1958.
[Cavendish Laboratory, Cambridge, and Metallurgy Division, AERE, Harwell, England]

A transmission electron microscope study of quenched aluminium revealed a high density of prismatic dislocation loops. These observations provided the first direct evidence for a mechanism in which the quenched-in vacancies precipitate into discs of vacancies, followed by collapse and shear, to form dislocation loops. [The *SCI*® indicates that this paper has been cited in over 260 publications.]

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The 1950s saw active research on the effects of supersaturations of vacancies and interstitials on the mechanical properties of metals and alloys, motivated by the need to understand radiation damage, of great importance to the development of nuclear reactors. While irradiation with energetic particles generates excess vacancies and interstitials at low temperatures, a metal quenched from a high temperature contains only excess vacancies, enabling vacancies to be studied separately, and thereby helping the interpretation of the more complex processes occurring in radiation damage. An important review by Cottrell¹ in 1957 showed that, while quench-hardening and resistivity studies gave information on the annealing kinetics of the quenched-in vacancies, it was not known whether these migrated to the dislocations (causing hardening by creating immobile jogs) or whether they formed clusters in the lattice (causing resistance to dislocation glide) as in precipitation-hardened alloys. No direct experimental evidence existed for such clustering.

In 1956 Hirsch, Horne, and Whelan² (in Cambridge) and Bollmann³ (in Geneva) demonstrated

that dislocations could be revealed at high resolution by transmission electron microscopy (TEM) of thin foils. In Cambridge, the Metal Physics Group were keen to apply this powerful new technique to as many unsolved problems in metal physics and metallurgy as possible, including quench hardening, while in Cottrell's group in the Metallurgy Division at Harwell, both Smallman and Westmacott, who had been searching quite independently for evidence for clustering using X-ray low-angle scattering experiments, recognised that the scale of the clustering was such that TEM might provide direct evidence.

Both groups succeeded in preparing suitable foils of polycrystalline aluminium, which was quenched from ~600° C into iced brine and subsequently thinned by electropolishing. The micrographs revealed a high density of unfaulted dislocation loops, 100 to 1,000 Å in diameter. Although image-contrast experiments to determine Burgers vectors were not carried out, the faulted Frank sessile type⁴ could be eliminated, and the loops were therefore identified as prismatic with Burgers vector $\frac{1}{2} < 110 >$, produced by shear of the original Frank loop on a {111} plane. These loops were of the type predicted by Kuhlmann-Wilsdorf,⁵ and some of them were observed to glide in the manner expected from their prismatic character. The quenched-in vacancy concentration corresponding to the total loop area was found to be ~10⁻⁴ per atom, as expected. Zones denuded of loops were observed at grain boundaries, demonstrating these to be vacancy sinks.

Cottrell, who became aware of the Cambridge experiments, suggested a joint publication. The importance of the paper was two-fold. First, it showed immediately that vacancies do cluster and that dislocation loops are formed, with important implications regarding annealing kinetics and hardening mechanisms. Second, it showed that TEM provided a powerful technique for studying defects on this scale, and this opened up a new and exciting field of study of the nature of point defect clusters in quenched and irradiated metals. Over the last 25 years or so, TEM has proved to be the major tool to study the structure of materials damaged by irradiation and to determine the processes of climb in materials at elevated temperatures. (For a recent review see reference 6.)

1. Cottrell A H. Point defects and the mechanical properties of metals and alloys at low temperatures. *Vacancies and other point defects in metals and alloys*. London: Institute of Metals, 1958. p. 1-39.
2. Hirsch P B, Horne R W & Whelan M J. Direct observations of the arrangement and motion of dislocations in aluminium. *Phil. Mag.* 1:677-84, 1956. (Cited 195 times.)
3. Bollmann W. Interference effects in the electron microscopy of thin crystal foils. *Phys. Rev.* 103:1588-9, 1956. (Cited 160 times.)
4. Frank F C. Sessile dislocations. *Proc. Phys. Soc. London A* 62:202-3, 1949.
5. Kuhlmann-Wilsdorf D. On the origins of dislocations. *Phil. Mag.* 3:125-39, 1958. (Cited 120 times.)
6. Eyre B L. Geometry and behaviour of prismatic dislocation loops and stacking fault tetrahedra. (Bilby B A, Miller K J & Willis J R, eds.) *Fundamentals of deformation and fracture*. Cambridge: Cambridge University Press, 1985. p. 369-84.