Figure 2 Highly-deformed ringed spherulite.

lite suggests a similar structure, i.e. purely radially oriented lamellar growth, whilst the deformation of the final structure is characteristic of the structure peculiar to polypropylene at lower crystallization temperatures [2]. If this is the case it would tie in with the idea of a crosshatched structure which could only be formed at low crystallization temperatures, hence giving rise to the different deformation characteristics in the lower temperature grown parts of the structure.

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J. L. WAY
J. R. ATKINSON
Department of Metallurgy
University of Leeds
Leeds, UK

Direct observations of dislocations in splat-cooled Co₅Pr

Direct observations of dislocations in splat-cooled Co$_5$Pr

Numerous attempts have been made to study crystal imperfections in metastable, rapidly quenched (splat-cooled) metal and alloy systems in an attempt to correlate their character and density with residual magnetic and/or super-conducting properties [1-4]. In many prominent systems, particularly cobalt-rare earth alloys, samples prepared by rapid quenching of a molten charge directed upon a low-temperature substrate are brittle, and possess shape and thickness inhomogeneities which render them unsuitable or difficult to prepare for transmission electron microscopy. While some samples can be prepared for observations of dislocations by

thin-film transmission electron diffraction contrast [5], it has not been possible to directly observe dislocations in such thin films by moiré fringes arising from the systematic translation or rotation of overlapping foils of sufficiently large crystalline areas [6-8].

In the present investigation, the fortuitous occurrence of superimposed, parallel layers comprising thin samples of Co$_5$Pr splat-cooled films gave rise to moiré patterns in the transmission electron microscope which allowed dislocations in various electron-transparent regions to be directly observed.

The initial Co$_5$Pr alloy was prepared in a conventional arc-furnace under argon, using 99.99 % Co wire and 99.9 % Pr ingots as starting materials. The residual reactive impurities were

removed by melting a titanium gettering button inside the furnace just before the sample itself was melted. Samples of Co_5Pr, prepared as 2 g buttons, were remelted and turned over 3 to 4 times in order to attain a homogeneous composition; and weight loss following homogenization was not greater than 0.5 %. The rapid quenching of these alloy buttons was then made in a purified argon atmosphere using a shock-tube splat-cooling apparatus as described by Duwez and Willens [9]. The samples were melted in this apparatus in a vycor crucible with a 2 to 3 sec induction pulse, followed by a 1400 psi helium gas-injection to atomize the melt, which impinged upon a room-temperature copper substrate through a small opening in the end of the crucible. The quenched specimens, consisting of tiny droplets flattened into varying thicknesses of foils upon the substrate, were removed in flakes, thinned lightly in a solution of equal parts of nitric, sulphuric, and phosphoric acids, and examined in a Hitachi Perkin-Elmer HU 125 electron microscope, operated at 125 kV.

Numerous observations of thin-film samples of Co_5Pr in the electron microscope revealed regions exhibiting prominently defined moiré fringes, which revealed the presence of dislocations in the overlapping sample areas. Fig. 1 illustrates some typical examples of these features. In Fig. 1a one can readily identify numerous dislocations, while Fig. 1b shows what appears to be a small stacking-fault segment, and additional, isolated dislocations.

Estimation of the dislocation density over a representative sampling of the Co_5Pr films examined resulted in a value of approximately 10^{10} cm^{-2}. This value might be compared with a value of approximately 5×10^8 cm^{-2} estimated from electron diffraction contrast images of dislocations in films of Co_5Sm prepared by liquid-phase sintering, ground to 5 mils (0.005 in.) thickness, and ion-thinned to electron transparency [5]. The higher value of dislocation density for the Co_5Pr would be expected simply because of the high cooling-rate, estimated to be within the range of 10^6 to 10^8 °C sec^{-1} [10]. In addition, the occurrence of closely-spaced dislocations in adjacent crystal planes as evident in Fig. 1 (noted specifically in Fig. 1b) would also appear to reflect upon the alloy response to the rapid quench. It is to be noted that condensed vacancy loops would arise in several instances of closely associated dislocations by a shift of one dislocation to an adjacent plane (Fig. 1b). This
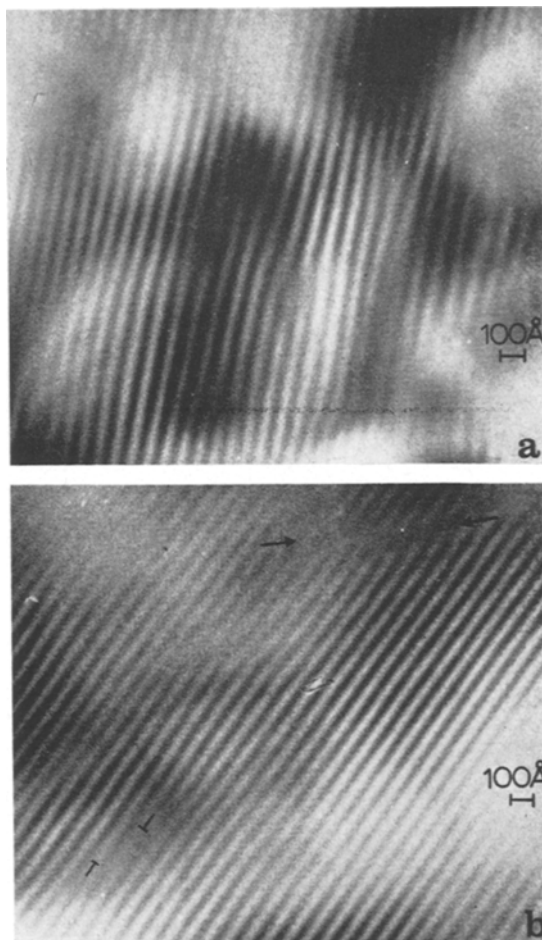

Figure 1 Dislocation moiré patterns in Co_5Pr splat-cooled films. (a) Numerous edge-type dislocation images in a layered-foil section. (b) Dislocations and a stacking-fault segment in a layered-foil section. Arrows denote the fault extension. Dislocations in the lower image portion are designated ⊥.

feature could possibly contribute to the high incidence of vacancies in splat-cooled alloys [1].

While the present observations of dislocations by moiré fringes in Co_5Pr have arisen somewhat fortuitously as a result of a layered sample morphology after splat-cooling, this feature could arise in other similarly prepared sample materials. Indeed, the recent work of Jones [3] suggests that any differential in cooling-rates during splat-cooling can give rise to layer solidification. More detailed investigations of residual defects in such alloy samples, using moiré techniques, could enhance our understanding of the metastable state in so far as crystal defects are concerned.

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H. P. SINGH
R. WANG
Department of Materials Science
University of Southern California
Los Angeles, California, USA
L. E. MURR
Department of Metallurgical and
Materials Engineering, New Mexico
Institute of Mining and Technology
Socorro, New Mexico, USA

Identification of copper precipitation in single crystal silicon using the CAMECA Ion Analyser

Infra-red microscopy can be used [1] as an indication of precipitation on crystallographic planes in copper-doped silicon; an example of such a micrograph is given in Fig. 1 where three families of precipitation on (1 1 0) and (1 1 1) planes can be seen intersecting the (1 1 1) surface of a silicon slice containing 10^{18} to 10^{19} atoms/cc of copper.

Identification of the precipitates as copper-rich material has now been achieved using a CAMECA IMS 300 Ion Analyser [2] with oxygen as the primary bombarding beam. Fig. 2 shows an image obtained in Cu^+ ions in the secondary ion spectrum from the surface of a sample similar to that used in the preparation of Fig. 1; three families of precipitation are again apparent and it is clear that the precipitates are copper-rich.
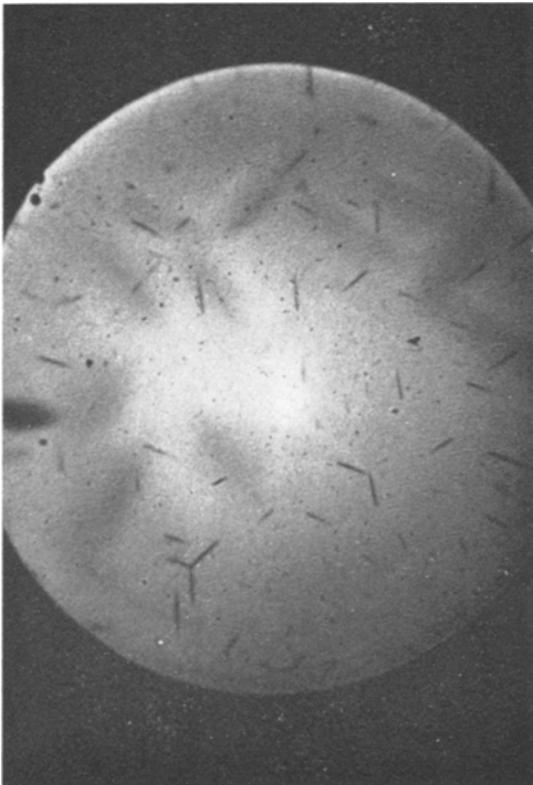
This example of the use of the ion analyser is

Figure 1 Infra-red micrograph of (1 1 1) silicon single crystal slice (× 275).

Figure 2 Cu^+ ion-analyser image of copper in (1 1 1) silicon slice. Primary O^+ beam (× 240).