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Determination of stress-strain curves of vapour-deposited films

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The strength properties of three vapour-deposited silver films, prepared with different thicknesses and crystal structures, have been compared by mounting them on electron microscope grids and then indenting them with a fine tipped stylus. The method is very rapid and enables specimens to be transferred to an electron microscope for microstructural studies, quickly and without risk of "handling" damage. A simple model of the indentation situation is proposed and this is used to calculate stress–strain curves for the films. Although the technique seems unsuitable for strains below $\sim 1\%$, very *large* strains can be reached, even when work-softening occurs. The results are consistent with those obtained on similar specimens by other investigators.

1. Introduction

Recent advances in vacuum vapour deposition technology have made it possible for thick coatings, [1-4] foils, [5, 6] and free standing parts [7] to be prepared. The versatility of the vapour deposition process allows a high degree of control of material structure and several instances have been reported in which materials have been prepared with mechanical properties superior to those found in conventionally made materials. For example, titanium alloy foils deposited by Smith *et al.* [5] were subjected to ductility and tensile tests and in both respects were superior to foil produced by conventional rolling methods. Dahlgren and Merz [2] deposited an iron 2% carbon alloy which had a hardness of 1240 Dph; this is as hard as the hardest cold-worked alloy steels. Structures apparently unobtainable by other methods have been prepared [2, 8]; little is known about the mechanical properties of such materials and clearly, further investigation is needed.

Vapour-deposited materials are most readily studied in thin film form. Specimen preparation is easy and microstructural effects can be studied directly by transmission electron microscopy. Thin film deformation has been investigated by several techniques; some of these are essentially qualitative [9, 10] whereas, in others, strength parameters are measured [11]. However, many of these techniques appear long and tedious and often there is a high risk of "handling damage" to the specimens. Usually, large areas of film are stressed and, in these cases, irregularities caused, for example, by cleavage steps or cracks in the substrate on which the film is deposited, can have an appreciable effect on the deformation.

A novel technique for studying the deformation of thin films has recently been developed [12-14]. Essentially, specimens are mounted on grids and indented, either quasi-statically or by impact. Here, it is shown that the technique allows rapid comparison of silver films prepared with different structures. Using a simple model of the indentation situation, stress-strain relationships for the specimens are obtained.

2. Experimental

Silver was chosen as a "test" material for the technique as this has been studied in thin film form by previous workers [11, 15-20]. Therefore, strength values obtained using indentation can be compared with those obtained by other methods.

Two sodium chloride crystals, cleaved on (100) were arranged symmetrically with respect to a molybdenum boat containing silver powder. One of the crystals was heated to 540 K; the other, which was coated with a thin layer of collodion plastic, was unheated. 250 nm of silver was deposited on the substrates at a rate of ~ 0.4 nm sec^{-1} in a vacuum of 3 mPa. The films were removed from their substrates by dissolving the sodium chloride in water and the collodion in acetone. They were then mounted on 3 mm, 200 mesh transmission electron microscope

TABLE 1

90 nm poly-crystal		250 nm poly-crystal		250 nm single-crystal	
Load, L (mN)	Depth, x (μm)	Load, L (mN)	Depth, x (μm)	Load, L (mN)	Depth, x (μm)
0.08	0.15	0.15	0.12	0.10	0.10
0.12	0.16	0.20	0.13	0.15	0.16
0.16	0.24	0.25	0.22	0.20	0.22
0.18	0.43	0.30	0.30	0.25	0.26
0.20	0.60	0.35*	0.42†	0.30	0.64
0.21*	0.75†			0.35	0.97
				0.38*	1.20†

*Average failure loads.
†Depth just prior to failure, estimated graphically.

grids, and annealed for 30 min at 920 K. Observation by transmission electron microscopy showed that the film deposited on the heated sodium chloride substrate was (100) single crystal and that deposited on the collodion was polycrystal. 90 nm polycrystal films were prepared by a similar procedure.

The apparatus used for loading was developed by Gane [21]. It consists of a stylus of tip radius 4 μm mounted on the arm of a galvanometer movement; loads are applied by varying the current through the galvanometer coil. The experiment is done inside a scanning electron microscope; this allows the point at which the load is applied to be selected and the deformation process to be studied at high magnification.

Loads were applied to the films in regions well away from the grid bars. Usually, three separate indentations were made at each load. Eventually loads were reached at which the films perforated. The average loads for perforation of each specimen were measured.

3. Results and discussion

The deformed specimens were studied by transmission electron microscopy. Fig. 1a shows a perforation in 90 nm annealed polycrystal film. Four tears, radiating outwards from the centre of the deformation site are produced. The petals between the tears are bent down by the stylus. This mode of perforation was also found with the 250 nm polycrystal and single-crystal films. The perforations are surprisingly similar in appearance to those found with (100) single crystal films in which tears are produced along ⟨110⟩ directions [13]. Dislocation tangles are seen for a distance ~ 200 nm from the edge of the tears in the film and this suggests that the tearing is a thinning process, taking place within the grains rather than at grain boundaries. Fig. 1d shows that the average grain diameter in the undeformed film is ~ 200 nm. Some of the grains are crossed by bands; these are probably growth twins.

The general appearance of the indentations formed was the same for each of the three specimens. Fig. 2 shows indentations in 90 nm polycrystal film. The shape of the indentation follows closely that of the stylus tip and there is little deformation outside the contact area. In Fig. 2h a small tear has initiated. As the indentations have a spherical form their depths can be estimated from their diameter. In Table I indentation depths produced by a series of applied loads are tabulated for the three specimens. The average rupture loads and the estimated indentation depths just before failure are also included in the Table.

The load-depth values in Table I enable rapid comparison between different films in a way similar to that allowed by the indentation test used on solid specimens. Several types of comparison are possible. For example, for films having the same thickness, the failure load and the load to produce a given indentation depth are indications of relative strength. The depth at which complete perforation occurs depends on the ductility of the specimen.

The stresses and strains in the indentation situation are estimated using the following analysis. It is assumed that the indentations have the form shown in Fig. 3. The specimen is deformed into a spherical cap by a pressure

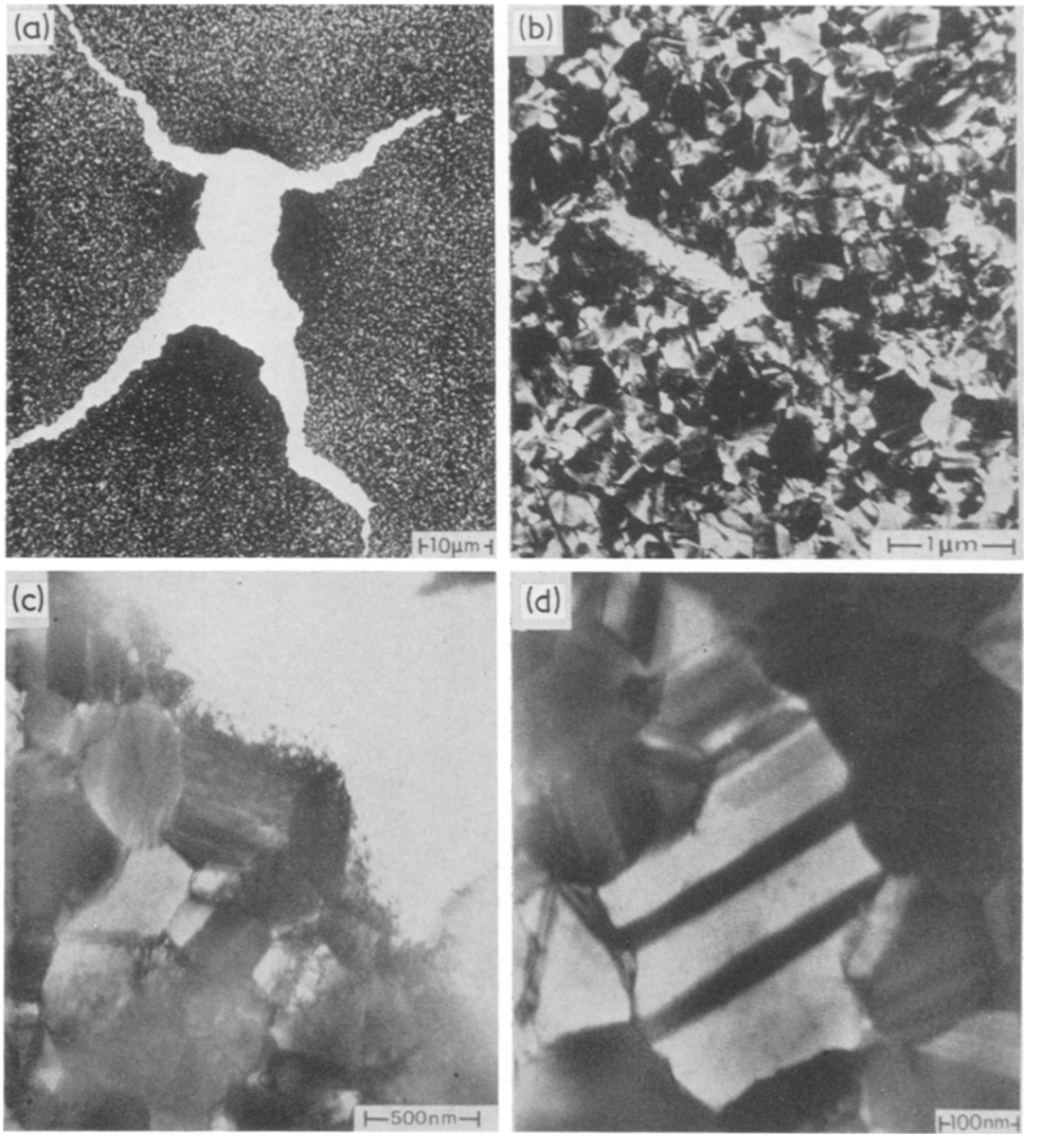

Figure 1 Transmission electron micrographs of a failure site in annealed 90 nm film. Dislocation tangles are seen at the edge of the tear in (c).

which may be taken as the load applied, L, divided by the area of contact between the stylus and the film, that is, as $L/2\pi ax$, where a is the indenter radius and x is the depth of the indentation. This situation may be compared with that in which a stress σ_s is produced in the wall of a hollow sphere of radius a by an internal pressure P. The stress σ_s is equal to $Pa/2t$ where t is the wall thickness. For the model considered (Fig. 3) $\sigma = 1/4\pi t \cdot L/x$, where σ is the stress in the film. The strain, ϵ, can be taken as the increase in area of the film divided by the original area in which this increase took place, that is $\epsilon = (2\pi ax - \pi r^2)/\pi r^2 = x/(2a - x)$ where r is the radius of the contact area.

Stress-strain curves obtained for the three films tested are shown in Fig. 4. It is seen that high strains are reached before failure occurs;

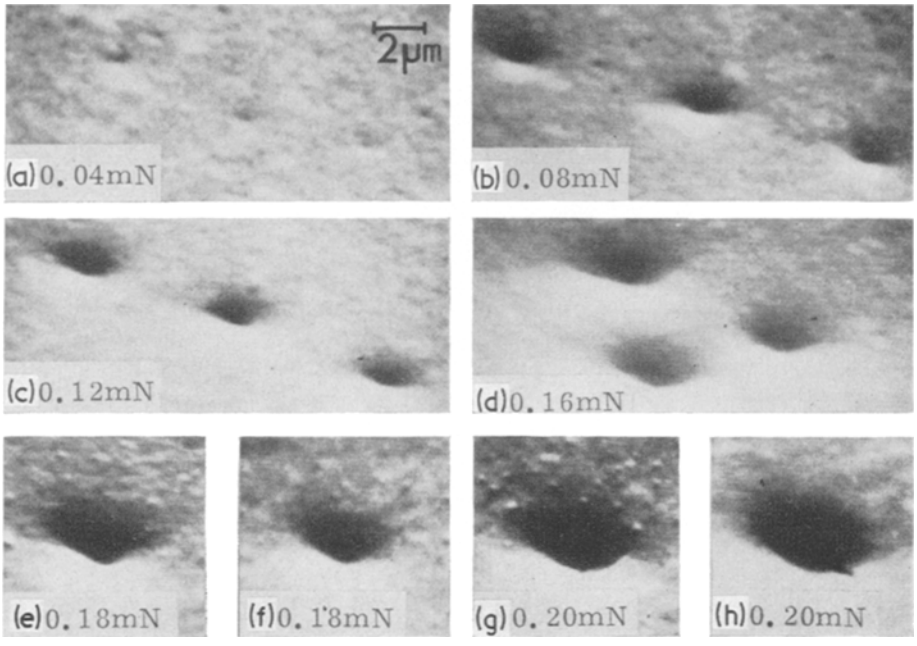

Figure 2 Stylus loaded 90 nm polycrystalline film; the loads applied are as indicated. A fissure is seen in (h).

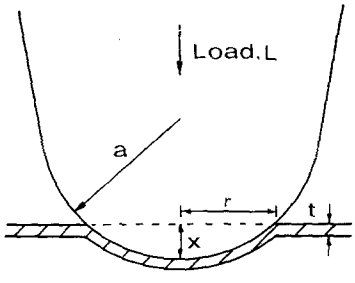

Figure 3 The model of the indentation situation, used to determine stress–strain relationships.

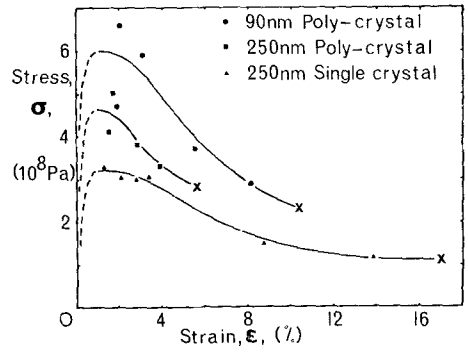

Figure 4 Stress–strain curves of the three films tested.

this is because the films do not necessarily fail when the flow stress begins to decline and therefore the "work-softening" part of the stress-strain curve is obtainable. The weakening at high strains is probably due to local thinning, producing fissures of the type seen in Fig. 2h. The technique appears to be unsuitable for strains below $\sim 1\%$ as these correspond to indentations which are very small and difficult to measure accurately. However, Beams *et al.* [15] obtained stress–strain curves of similar specimens for small strains and the likely shape of the complete curve can be estimated by using these curves (shown broken in Fig. 4), to extend to low strains those obtained here by indentation.

The flow stresses obtained here are comparable with those found by previous workers. For example, Beams [19] used a bulge test to stress polycrystalline silver and found that films thicker than $\sim$ 100 nm had a tensile strength $\sim 2 \times 10^8$ Pa. Working on similar specimens, Palatnik *et al.* [18] used electron diffraction to estimate the elastic strain at failure; the tensile strength inferred was $\sim 4 \times 10^8$ Pa. Weinstein *et al.* [16] obtained values ranging from 1.6×10^8 to 3.3×10^8 Pa. Further, using the indentation technique it is found that 90 nm polycrystal film is stronger than 250 nm film and that (100) single crystal silver is weaker, though more

ductile than polycrystalline silver. Both these results are consistent with other workers' findings. See, for example, the work of Beams [19] and Weinstein *et al.* [16] on the effect of thickness on strength and Blakely's [20] comparisons of (100) single crystal with polycrystal gold films.

4. Conclusions

Thin films of silver have been mounted on electron microscope grids and indented in the unsupported regions between the grid bars. It is shown that the technique can be used to rapidly compare the strength properties of vapour deposited materials and, at the same time, allows deformation mechanisms to be studied by electron microscopy. An analysis based on a simple model, is used to obtain stress–strain curves. The technique is effective even in situations in which the flow stress declines with strain and, as a result, flow stresses can be measured at very large strains. It is suggested that the work-softening observed with the silver films tested occurs as a result of localized thinning of the specimen.

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