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VAKIL SINGH
P. RAMA RAO
*Department of Metallurgical Engineering,*
*Banaras Hindu University,*
*Varanasi,*
*India*

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## *Transmission electron microscopy and X-ray analysis of coloured anodized films prepared by ultramicrotome without embedding*

Direct investigation of thin anodized and subsequently electrolytically coloured oxide films with a high resolution is only possible if a method is used which is not selective during the decomposition of different materials. During ion-bombardment, this requirement is not fulfilled [1], therefore ion-beam etching, proposed earlier [2], should be avoided in this case. Furthermore, all treatments having an effect on the sample (including embedding [3]) should be avoided to be sure not to influence the colouring agent. A very simple method to provide thin sections is described below, and the interpretation of structures observed in this way is given.

In order to prepare thin sections for direct examination, anodic layers were prepared on 5 mm thick sheets of aluminium. From these sheets 5 mm wide and 15 mm long strips were cut, the strips were trimmed and then cut with the ultramicrotome. The same procedure was carried out in the case of layers built up on bulk material. Blocks with dimensions 5 mm × 5 mm × 15 mm were self-

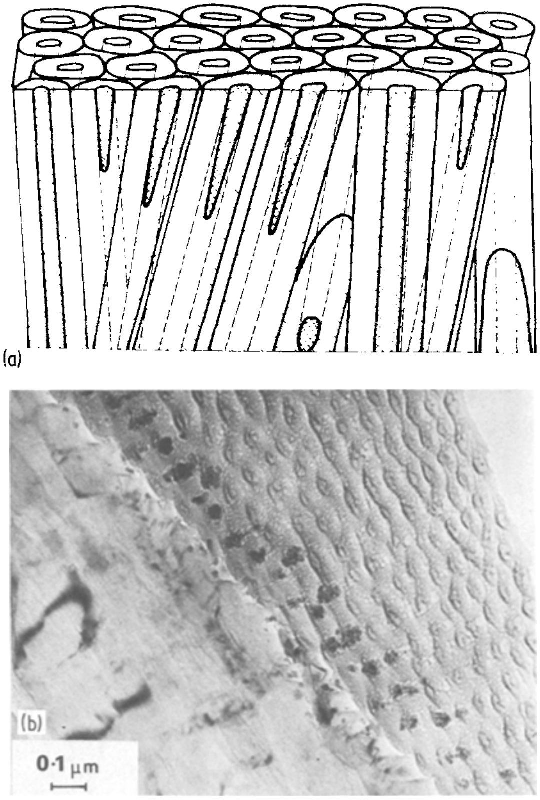

*Figure 1* (a) Schematic model of a section. (b) Micrograph of a section inclined to the surface. Anodic layer coloured for 20 sec.

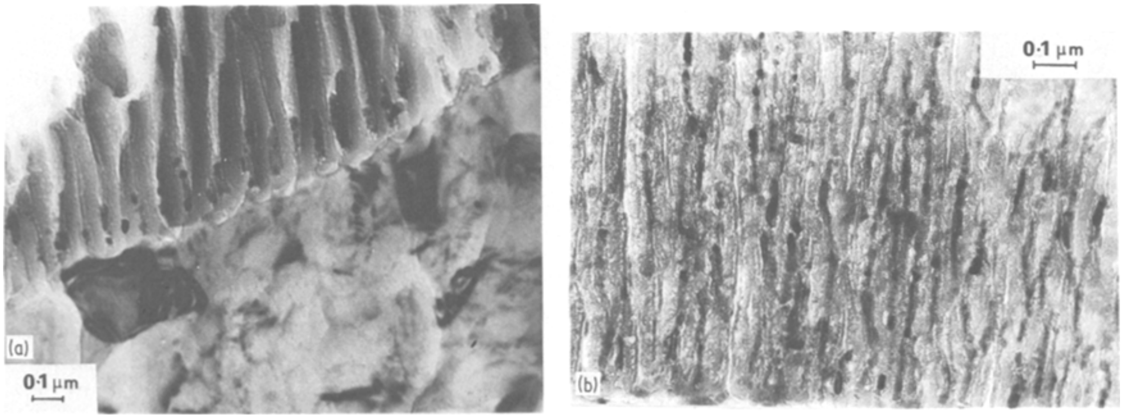

*Figure 2* (a) Vertical section of an anodic layer coloured for 20 sec. (b) Vertical section of an anodic layer coloured for 5 min.

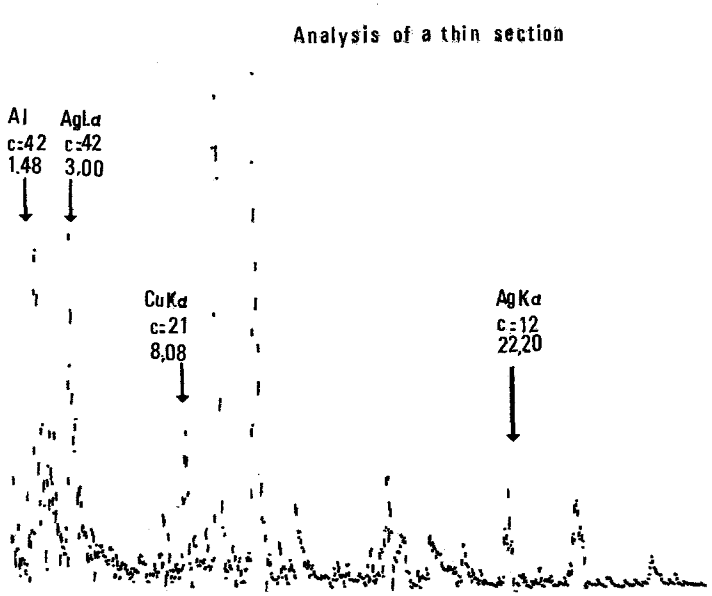

*Figure 3* Energy dispersive analysis of a single silver colouring agent column.

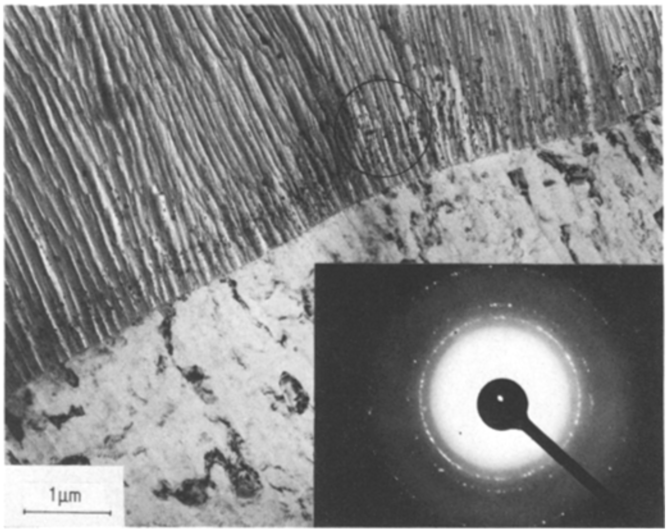

*Figure 4* Micrograph and polycrystalline diffraction pattern of the copper colouring agent.

supporting, i.e. strong enough without embedding. To avoid misinterpretations of structures prepared in this way, anodic layers made by different methods without colouring were also studied [4]. Finally, the coloured layers themselves were studied: the layers were coloured with different solutions for different times [5].

It is well known that anodic oxide layers are amorphous when investigated by electron or X-ray diffraction, but have at the same time a periodic structure consisting of cell prisms and of pore-channels free of oxide in the axis of the cells [6]. It could be directly observed that during the colouring process, metal is deposited into these pore channels.

In the sections (thickness ~10 to 100 nm) the oxide cell-pore structure can be observed along different planes of sectioning, as the blade and sample cannot be exactly oriented to each other and the micro-orientation of the oxide changes within a single preparation. With this method, taking the thickness of the sections into consider-

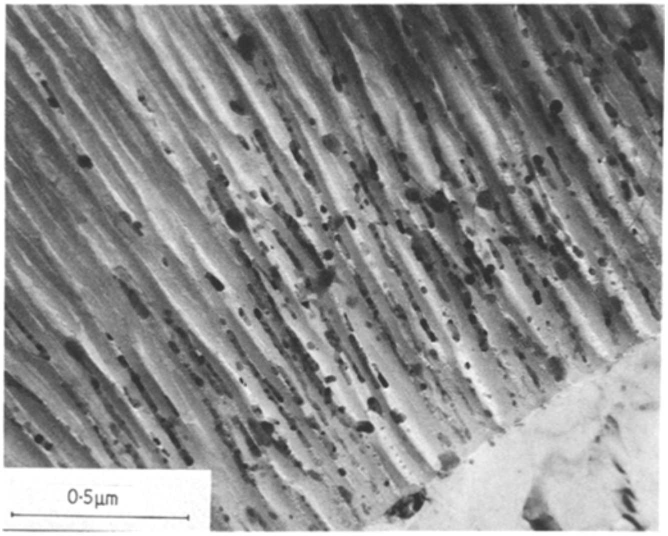

*Figure 5* Micrograph of an anodic layer coloured with two different metals for 3 min.

ation, the colouring metal rods having the same diameter as the pores are also sectioned. This is shown in Fig. 1a and b.

If our sections are true vertical sections, the method is suitable for studying the effect of colouring time as shown in Fig. 2a and b. Our method of investigation is able to provide the answers to the following questions: (1) what chemical and physical state of the metal is responsible for the colouring effect as seen in Figs. 3 and 4, and (2) what is the directly presented spatial distribution of the different colouring agents in the layer as shown in Fig. 5?

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Á. CSANÁDY
*Research, Engineering and*
*Prime Contracting Centre,*
*Hungarian Aluminium Corporation,*
*Budapest,*
*Hungary*

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## *Chemical instability of CaO:Y phosphor — a thermoluminescence study*

Thermoluminescence (TL) of CaO phosphor doped with various suitable activators has been studied by several research workers, reference to which can be found in the papers of Schwartz and Janin [1] and Khare [2]. It is well known that CaO is relatively unstable in air; reacting with atmospheric moisture it forms $Ca(OH)_2$. Recently, Lehmann [3] has pointed out that because of the problem of chemical stability of CaO in the open atmosphere, although some of the suitably activated CaO phosphors are efficient, their technical utility is questionable. Lehmann's [3] observation is based on cathodoluminescence studies. Incidentally, it is to be noted that in most of TL studies during the excitation/irradiation of the phosphor, in addition to heating the phosphor while recording TL, the sample is invariably exposed to atmospheric moisture. However, in the earlier studies of the TL of CaO