#565

(*Reprinted from Nature, Vol. 206, No. 4986, pp. 816–817, May 22, 1965*)

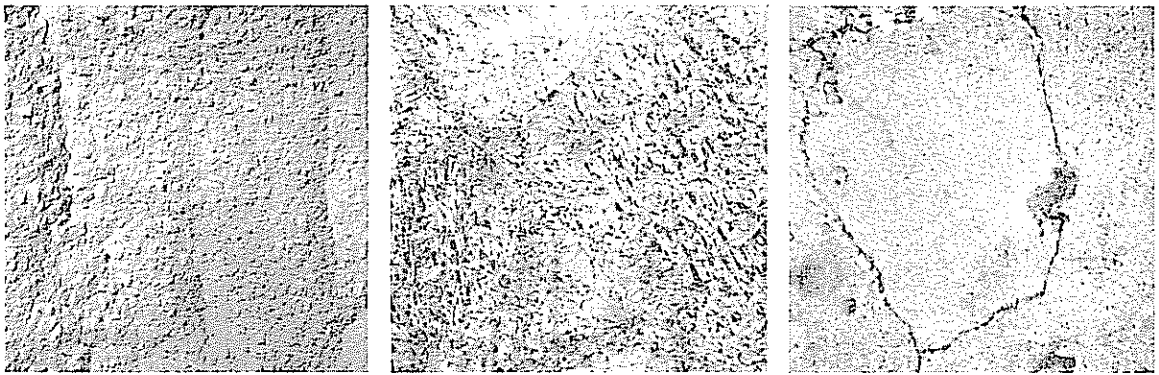
Effect of Heat Treatment on the Structure of Oriented Nickel Electrodeposits

INVESTIGATIONS on the development of preferred orientations on electrodeposits using electron diffraction techniques[1–4] and examination of texture and topography of electrodeposited metal surfaces[4–8] have been reported in recent years. Little information is available at present, however, regarding the effects of heat treatment of electrodeposited metals, at different temperatures, on changes in the foregoing properties. An electrodeposited metal develops not only preferred orientation but also internal stresses[9–11], their extent depending on deposition conditions. Since annealing removes most of the stresses in a strained metal, it is of interest to follow possible changes accompanying such treatment.

Nickel can be formed by electrodeposition not only in a state of high purity but also with widely different preferred orientations[12]. Initial results are reported in this note on the changes in texture of electrodeposited nickel, resulting from thermal treatments at or above its recrystallization temperature.

Nickel was electrodeposited at a current density of 10 m.amp/cm^2 for 25–30 min on surfaces of 2-mil thick polycrystalline nickel foil at a bath temperature of 25° C, unless otherwise stated. The thickness of the deposits, in most cases, was about 5μ. The deposition was made from sulphate–chloride baths (Watt's type), which were previously purified by prolonged low current electrolysis using a dummy cathode, followed by repeated filtration. All the chemicals including the nickel foil were of reagent grade. The nickel cathode surface was initially cleansed by etching in conc. hydrochloric acid, followed by washing with distilled water. The freshly prepared deposits were first examined with 50 kV reflexion electrons followed by heat treatment in a helium atmosphere as described here.

The recrystallization temperature of nickel ranges from 530° to 600° C (ref. 13). In order to examine the changes in texture of electrodeposits occurring around the recrystallization temperature, heat treatments were carried out in an ultra-dry helium atmosphere at: (1) 450° C for 60 min; (2) 550° C for 60 min; (3) 600° C for 30 min. The rate of heating up to and cooling down from maximum temperature was about 3° C/min.



a *b* *c*

Fig. 1. Electron micrographs of the nickel surfaces obtained by electrodeposition from a bath of *p*H 3·9. (*a*) Original surface (× *c.* 3,100); (*b*) heated to 550° C (× *c.* 6,000); (*c*) heated to 690° C (× *c.* 2,050)

Table 1. Effect of Heat Treatment on Orientation of Electrodeposited Nickel

Original orientation	Heat treatment at °C		
	450	550	690
(210)	(210)	*r*	*r*
(100)	*s*(111) + *m*(100)	*s*(111) + *m*(100)	*r*
(10$\bar{1}$0) + (211)	*nr*	*nr*	*r*
(110)	*w*(110) + *m*(311)	*r*	*r*

r = random; *nr* = nearly random; *w* = weak; *m* = medium; *s* = strong.

The heat-treated samples were identified structurally by evaporating 50-Å thick platinum on parts of their surfaces and measuring the d_{111} diffraction line of platinum as an internal standard, along with *d*-spacings for the heat-treated nickel by means of reflexion electron diffraction. In order to follow the changes in topography of some selected metal surfaces resulting from heat treatment, platinum pre-shadowed carbon replicas of the surfaces were studied by electron microscopy[4].

Nickel, electrodeposited on surfaces of clean and polycrystalline nickel foils, develops a random orientation during the early stages of deposition up to a certain thickness, when a bath of *p*H 2·1 is used[1,3,12]. For longer times of deposition, these bath conditions favour the development of a (210) preferred orientation. Electrodeposition from baths of *p*H values of 3·9 and 5·1 favours the development of (100) and (10$\bar{1}$0) + (211) preferred orientations, respectively, at advanced stages of deposition. The development of mixed structures (that is, hexagonal close packed (10$\bar{1}$0) + face-centred cubic (211)) in electrodeposited nickel under certain conditions of deposition has been discussed in detail in the past[12,14]. Any of the foregoing baths when maintained at 75° C favour the development of a (110) orientation, during advanced stages of deposition.

Table 1 summarizes the results as to the effect of heat treatment on the orientation of the nickel electrodeposits, as determined by electron diffraction. Similar changes in diffraction patterns during heat treatments are also found to occur if the oriented nickel deposits are formed on polycrystalline platinum foils.

Fig. 1*a* shows the topography of the electrodeposited nickel surface (5μ thick), developing (100) preferred orientation. Figs. 1*b* and *c* show nucleation and grain growth occurring in the deposits as a result of heat treatment at 550° and 690° C respectively.

Our results on the heat treatment of oriented nickel deposits thus reveal that whatever their initial texture or preferred orientation, heating the specimens in a neutral atmosphere, in the temperature-range where recrystallization of nickel is known to occur, leads to a structure which shows random orientation by reflexion electron diffraction. It is thus seen that in adsorption and catalysis investigations which use nickel films, one should be concerned about the temperature history of the film being used.

Frequently, the assumption is made that the (100), (110) and (111) planes are equally exposed in the surface.

This work was supported by U.S. Atomic Energy Commission on contract *AT*(30–1)–1710.

B. C. Banerjee
P. L. Walker, jun.

Fuel Technology Department,
Pennsylvania State University, University Park.

[1] Finch, G. I., Wilman, H., and Yang, L., *Dis. Faraday Soc.*, **1**, 144 (1947).
[2] Finch, G. I., and Layton, D. N., *J. Electrodepositors Tech. Soc.*, **27**, 215 (1951).
[3] Banerjee, B. C., and Goswami, A., *J. Electrochem. Soc.*, **106**, 20 (1959).
[4] Banerjee, B. C., and Walker, jun., P. L., *J. Electrochem. Soc.*, **108**, 449 (1961).
[5] Pick, H. J., Storey, G. G., and Vaughan, T. B., *Electrochim. Acta*, **2**, 161 (1960).
[6] Barnes, S. C., *Electrochim. Acta*, **5**, 79 (1961).
[7] Weil, R., and Cook, H. C., *J. Electrochem. Soc.*, **109**, 295 (1962).
[8] Economou, N. A., Fischer, H., and Trivich, D., *Electrochim. Acta*, **2**, 207 (1960).
[9] Newell, I. Laird, *Metal Finishing*, 56 (October 1960).
[10] Brenner, A., and Senderoff, S., *J. Res. Nat. Bur. Stand.*, **62**, 89, 105 (1949).
[11] Phillips, W. M., and Clifton, F. L., *Proc. Amer. Electroplaters Soc.*, **36**, 97 (1947).
[12] Banerjee, B. C., and Goswami, A., *J. Sci. Indust. Res.* (India), **14**, B, 322 (1955).
[13] Wilman, H., *Proc. Phys. Soc.*, **68**, B, 474 (1955).
[14] Yang, L., *J. Electrochem. Soc.*, **97**, 241 (1950).

Printed in Great Britain by Fisher, Knight & Co., Ltd., St. Albans.