

sharply at 31°. Calcd. for $C_6H_9O_2N$: C, 56.6; H, 7.1. Found: C, 56.55; H, 7.2.

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FREDERICK C. B. MARSHALL

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COMMUNICATIONS TO THE EDITOR

THE CRYSTAL STRUCTURES OF ELECTRODEPOSITED ALLOYS. SILVER-CADMIUM

Sir:

The crystal structures of silver-cadmium alloys, ranging in composition from 20% Cd to 96% Cd, have been obtained from x-ray diffraction data. The alloys were deposited at room temperature according to the method of Stout [Preprint No. 29, *Trans. Am. Electrochem. Soc.*, **59** (1931)], using a current density of one ampere.

In general, the structures of the alloys are quite different from those of thermal alloys of corresponding composition which have been brought to equilibrium before examination. The alloys prepared under equilibrium conditions [Astrand and Westgren, *Z. anorg. allgem. Chem.*, **175**, 90 (1928)] show the following phases.

Percentage Cd

0-44	α -solid solution of Cd in Ag
44-49	$\alpha + \beta$
49-51	β - C_2Cl type cubic lattice. β' -close-packed hexagonal lattice, similar to ϵ , obtained when β is heated above 400° and suitably quenched. It differs from ϵ in axial ratio
51-57	$\beta + \gamma$
57-66	γ -body-centered cubic lattice
66-69	$\gamma + \epsilon$
69-83	ϵ -close-packed hexagonal lattice
83-95	$\epsilon + \eta$
95-100	η -solid solution of Ag in Cd

Westgren notes that β should also be formed at high temperatures in the γ -range but his attempts to produce it by heating the γ -phase failed.

The electrodeposited alloys show the following structures: (1) 40% Cd—contains the α , β' and γ phases and may contain the β phase; (2) 46-75% Cd—only the ϵ phase is deposited. The crystals show a preferred orientation with respect to the base metal, and this orientation differs with the composition; (3) 89-96% Cd—the ϵ and η phases are deposited.

Our results do not agree with microscopic data recently published by Fink and Gerapostolou [*Metal Ind.* (N. Y.), **28**, 519, 562 (1930)].

It is evident that under the conditions of deposition used in these experi-

ments the ϵ -phase is produced throughout the greater part of the range, and is apparently a metastable phase at least through the approximate range 46–66% of Cd. This is consistent with the behavior of some pure metals, which are known to be electrodeposited in metastable forms.

It is important to know how the conditions of deposition affect the crystal structure of an alloy of any given composition, since hardness and other physical properties depend upon the structure.

We are at present investigating (1) the effect of current density and temperature on crystal structure, the composition of the deposit remaining constant; (2) the recrystallization temperatures of Cd–Ag alloys.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF ILLINOIS
URBANA, ILLINOIS
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CHARLES W. STILLWELL

EXPLOSION DURING THE CATALYTIC REDUCTION OF NITROANISOLE IN THE LIQUID PHASE

Sir:

Brown, Etzel and Henke [*J. Phys. Chem.*, **32**, 631–635 (1928)] quantitatively reduced a number of nitro compounds in the liquid phase at about 215°, under a hydrogen pressure of about 500 lb. per sq. in., using a nickel catalyst. The reduction could be made either without a solvent or with benzene and ethyl alcohol. Their work was done on small quantities (about 5 g.) of material, in a shaking autoclave maintained at the desired temperature by immersion in an oil-bath.

The writer attempted to apply this method to the reduction of *o*-nitroanisole. As no danger was anticipated, the work was done on a *technical* scale, using 400 g. of the nitro compound. A shaking autoclave of steel was used, of about 3000 cc. total capacity, heated electrically. The first experiment was made using benzene as a solvent, with a nickel catalyst, and gave approximately a 50% yield of amine. In the next, the solvent was omitted and the catalyst was carefully prepared to give a very active product; 250 g. of nickel nitrate was ignited at 400°, and then heated for three hours in a muffle at 560°. The oxide was reduced in the autoclave under a stream of hydrogen at a temperature of 280–300°. Four hundred grams of the nitroanisole, freshly distilled, was then added and hydrogen was passed in to a pressure of 500 lb. At a temperature of 250°, with shaking, the hydrogen was rapidly taken up, and the pressure dropped in about fifteen minutes to 250 lb. It then began to rise rapidly, and reached a total of 500 lb. in about five minutes. In thirty seconds more the pressure jumped to 1000 lb. The writer then opened the needle valve on the autoclave in the hope of relieving the pressure. Gas and liquid shot out of the valve, and an instant later the autoclave exploded with great violence.