Detection of α-Phase Precipitates in Decomposing NiH_β by Bitter Powder Pattern

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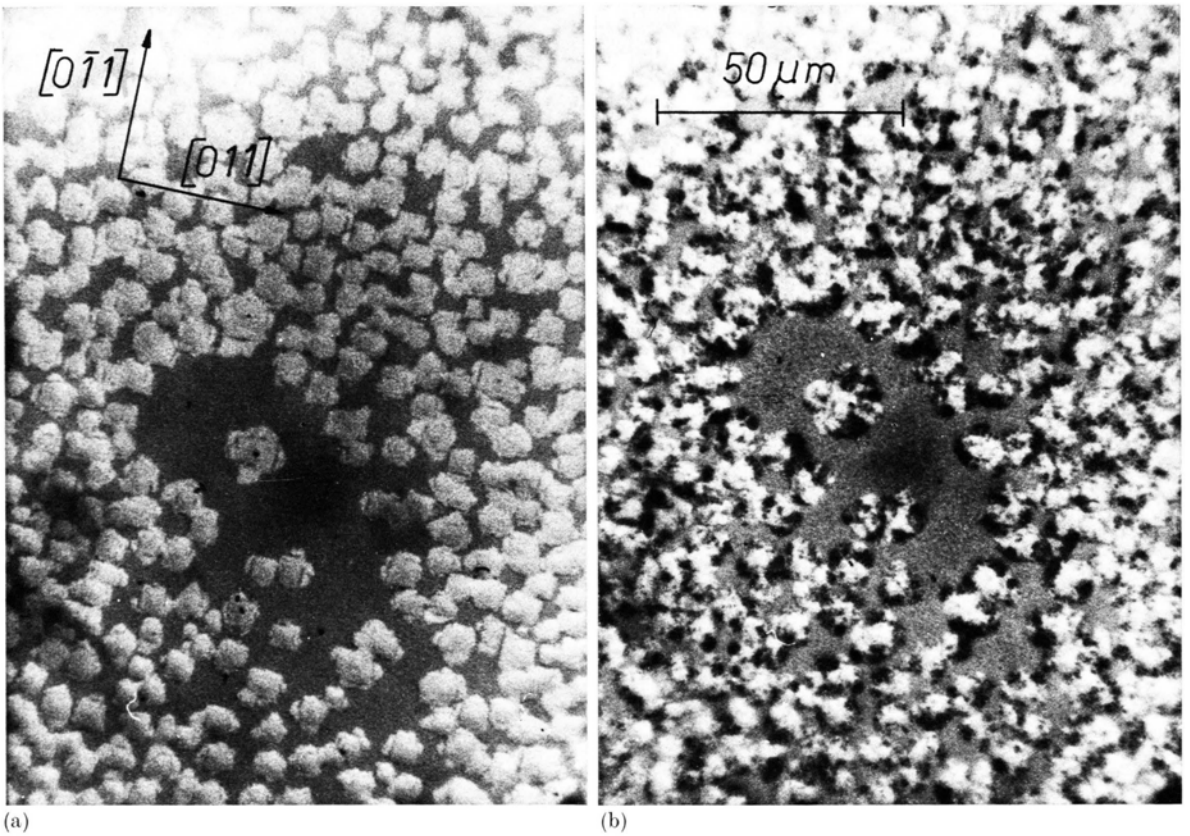
The application of a decoration technique basing on the settlement of iron single domain particles made it possible to identify directly small ferromagnetic α-phase particles in early states of decay of nickel hydride. In crystals of different orientation of the surface the shape and orientation of particles could be defined.

In a preceeding paper [1] we proved that a variation of the well-known Bitter technique according to Hutchinson [2] is appropriate to study transformations in which a ferromagnetic component comes into existence. We were able to show the possibility how to apply this technique on the analysis of metal-hydrogen systems, especially of those with the ferromagnetic component nickel.

In the case of the non-ferromagnetic nickel hydride [3] (NiH_β, with atomic number ratio H/Ni between 0,7 and 1) the desorption of hydrogen gives rise to the ferromagnetic α-phase (with H/Ni between 0 and about 0,01), which has the spontaneous magnetization of pure nickel. This circumstance — i.e. the coexistence of magnetic and nonmagnetic regions — allows the application of the Bitter technique to recognize an arrangement of α- and β-phase areas in the surface or in layers next to it. The Bitter method was chosen because such a proof is hardly disturbed by surface roughness; its modification according to Hutchinson furthermore is apt to produce a better contrast of areas.

The Bitter method means that the surface of the sample is decorated with small ferromagnetic



(a) (b)

Fig. 1. Optical micrographs showing the same part of the (100)-surface in only slightly different α + β-states of desorption of β-NiH; (a) before, and (b) after decoration with iron particles.

particles. In the modification according to Hutchinson which we used, the particles are produced by evaporating iron in an atmosphere of argon. Immediately after their condensation in this atmosphere they are attracted by the stray-fields of the sample and do finally adhere to its surface. Accumulations can be recognized with an optical microscope in vertical illumination as blackening.

The hydride was obtained by cathodic charging [4] of electrolytically polished nickel discs at room temperature. During the charging process with a relatively flat transformation front α/β penetrating into the crystal [5, 6], the surface remained optically smooth.

Figures 1 a and 1 b represent optical micrographs of the (100)-surface in two only slightly different states of decay: On the originally uniform surface irregulary distributed lighter islands have appeared. Some of them show the square shape (Fig. 1 a), which in earlier states of desorption is typical of even more of these small areas. In order to prove directly the precipitation-like character of the desorption, which can be deduced from previous magnetic investigations [7, 8], the magnetic state of the sample was revealed by the Hutchinson technique: In Fig. 1 b it can be seen that only within the light islands (now enlarged by further desorption) dark spots of Bitter Powder were formed. This means ferromagnetism, i.e. α-phase in dispersed form, embedded in the even decorated non-ferromagnetic β-phase.

An additional information is given by the fact that the small ferromagnetic areas are not decorated evenly. This effect is caused by the internal strains which result from the 15% volume decrease accompanied by the phase transformation $\beta \rightarrow \alpha$. The strains are large enough to determine the directions of easy magnetization, and thus states of recovery in the course of desorption can be made visible by powder pattern.

The preferred plane for precipitation is always that {100}-plane which is at the smallest angle to the surface. In the case of a {100}-surface as shown in the figure, the surface itself is the plane of precipitation, which means that the platelets of NiH_α which came into existence, are parallel to the surface and therefore the microstructure is least damaged by the desorption process. In general the shape of the precipitates is needle-like with the trace of the according {100}-plane as the long axis.

The example shows that the application of this method produces by ferromagnetic marking also under complicated conditions definite suggestions about the mechanism of decomposition.

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