

ARTEFACTS INTRODUCED BY ELECTROPOLISHING INTO Fe-Ni BASED AUSTENITIC ALLOYS

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It was found in investigating of the structural states in austenitic Fe alloys with 19% Ni and 10% Ru; 27% Ni and 2% Ru, and in an 34.4% Ni with FCC lattices that particles of a solid Fe-Ru solution in NiO form in the course of electrical polishing. No such particles were observed in a Fe alloy with 20% Ni, which has an α phase structure with a BCC lattice. The formation of the (Fe, Ru, Ni)O particles is attributed to penetration of oxygen atoms into the austenite lattice.

The change over from the replica technique to direct methods of electron-microscopic study with thin foils has resulted in the development of a new electron diffraction microscopy. The results obtained with thin foils are also valid for massive specimens, provided that the test specimens of foils are prepared in conformance to rules [1]. Basically these rules require that all heat treatment procedures are performed with massive specimens, after which they are thinned out, first mechanically and then electrolytically. However, electrical polishing has, in certain alloys, most probably as a result of stress relaxation on free foil surfaces, resulted in transformations and formation of new phases, characteristic only of thin foils, and not occurring in massive specimens [2]. Moreover, investigators have warned to be careful concerning new phases detected in thin foils, since electrical polishing may produce so-called artefacts (false structures) into the soil structure [1].

We have encountered such a situation in investigating the structural states of alloys of iron with 19% Ni and 10% Ru, 27% Ni and 2% Ru, and of an alloy of iron with 34.4% Ni. Thin foils of the alloys, polished mechanically to 0.03 mm were subsequently polished electrically at room temperature and 20 V in electrolyte, consisting of a saturated solution of chromic anhydride in orthophosphoric acid.

The electron diffraction patterns of all the austenitic alloys with a FCC γ lattice and a between 3.60 and 3.58 Å always exhibited additional reflections in the form of intense arcs, located about all the matrix reflections at the direction of small reflection angles (Fig. 1). These arcs correspond to reflections from the FCC lattice with $a = 4.19$ Å. The dark-field image, obtained in the reflection of an arc (Fig. 2) clearly depicts fine particles about 2000 Å in size. The orientation of particles coincides with that of the matrix, but the highly texturized reflections from them point to some scatter in their orientation.

The table lists experimental values of interplane distances d , corresponding

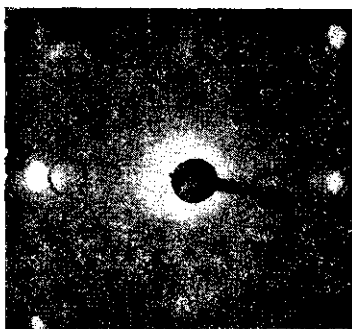


Fig. 1. Electron diffraction pattern of an alloy of iron with 27% Ni and 2% Ru. The electron beam is parallel to the [100] direction.

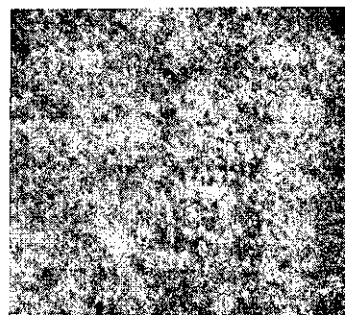


Fig. 2. Dark-field microphotograph of an alloy of iron with 27% Ni and 2% Ru, obtained in the reflection, designated by a circle in Fig. 1.

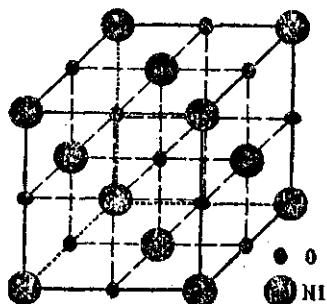


Fig. 3. Structure of NiO.

No	d_{exp} , Å	d_{table} , Å	hkl
1	2,42	2,41	111
2	2,07	2,09	200
3	1,47	1,48	220
4	1,25	1,26	113
5	1,19	1,20	222
6	1,04	1,04	400
7	0,96	0,96	331

to arcs; these values of d coincide with the tabulated data for NiO, which has a type NaCl type lattice with $a = 4.19$ Å (Fig. 3).

It is interesting to note that no arcs were observed on the electron diffraction patterns of an alloy of iron with 20% Ni, which has an α phase structure with a BCC lattice and $a = 2.86$ Å.

The above result is easily explained. In fact, in order for an NiO lattice with a type NaCl structure to form, it suffices, without changing anything in the austenite lattice, to fill all the octahedral voids of this lattice by oxygen atoms. Evidently, this occurs in the course of electrical polishing. Here the lattice parameter increases from 3.60 to 4.19 Å. In fact, a solid solution of Fe and Ru in NiO, i.e., (Fe,Ru,Ni)O, forms in the Fe-Ni-Ru triple alloy. The formation of a type NaCl lattice from the BCC structure of the α phase requires not only penetration of oxygen into the matrix lattice, but also must be preceded by restructuring of the BCC into an FCC lattice, which naturally does not occur during electrical polishing.

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