

## Orientation relation between Ni and NiO and its evolution during oxide growth

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Nickel is a reference material for fundamental studies on high temperature oxidation of metals. Numerous studies have been devoted to the Ni/NiO system, including the effect of nickel surface crystallographic orientation on oxidation kinetics and on oxide scale microstructure.

The use of nickel single crystal samples has allowed Khoi et al [1] to quantify the effect of crystal orientation on the oxidation kinetics. These authors have shown that the parabolic constant at 500°C is one order of magnitude higher for Ni (100) surfaces than for (111) surfaces. Later studies have confirmed this observation.

Recent works have shown that nickel surface orientation strongly influences the thickness of the oxide scale formed, but also its microstructure [2-4]. This was observed using polycrystalline samples with coarse and stable grains obtained after Ar-H<sub>2</sub> annealing during 30 minutes at 1350°C. The effect of surface orientation was confirmed at lower temperatures (500°C) but was also observed for 10 micrometers thick scales formed at higher temperatures, up to 1200°C.

The same kind of polycrystalline nickel sample is used in the present work in order to determine the orientation relationships between the metallic substrate and the oxide scale and to follow the evolution of nickel oxide texture with time.

A polished and annealed nickel sample was first observed in a SEM with an EBSP system in order to determine the orientation and the coordinates of eleven individual nickel grains. This sample was then oxidized under flowing oxygen at 700°C in a thermobalance. The oxidation was stopped for a mass gain corresponding to an average oxide scale thickness of about one micrometer. The cellular morphology [2] of nickel oxide scale was observed with the SEM. Nevertheless, this morphology depends strongly on the nickel grain surface orientation (figure 1).

X-Rays diffraction texture analysis was then performed for each nickel grain by using a mask with a 2 mm diameter hole. The XRD measurements allowed to determine the orientation of each nickel grain and the texture of the oxide scale grown on the surface of each of these nickel grains.

This procedure was repeated a second time after a second oxidation of the same sample at 700°C, up to an oxide scale thickness of about 6 micrometers.

The experimental procedure used in this work permitted to determine the crystallographic orientation relations between the substrate large nickel grains and their oxide grains. This could be done for several nickel grains with no special surface orientation. For an average oxide scale thickness of one micrometer and six micrometers, the cube on cube epitaxial relationship was

found between Ni and NiO :

$\{111\}\text{Ni} // \{111\}\text{NiO}$  and  $\langle 110 \rangle \text{Ni} // \langle 110 \rangle \text{NiO}$

Only for one nickel grain, the  $\langle 110 \rangle \text{Ni} // \langle 112 \rangle \text{NiO}$  was found. These results are compared with previous classic texture analysis [5-6]. The consequences of these observations on the oxidation kinetics are discussed.

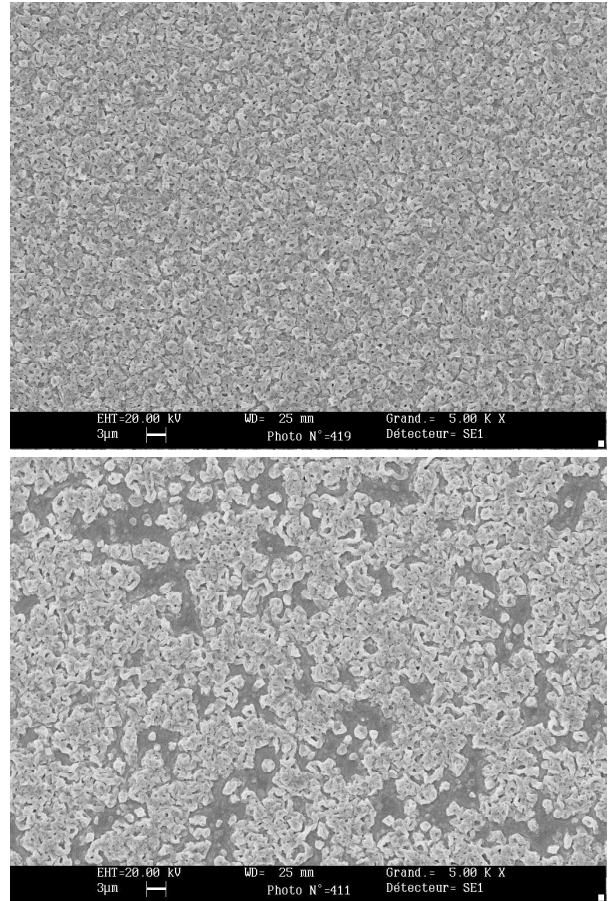


Figure 1. : NiO oxide scale formed at 700°C on two nickel grains with different crystallographic orientations. Both oxide morphologies are cellular but with different density (one micrometer average oxide thickness).

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