1464
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Preparation and Characterization of Pt/γ-Al_2O_3 Model Catalyst on NiAl Alloy

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Numerous studies of heterogeneous catalysis systems clearly demonstrate that the metal nanoparticle (NPs)/support interaction is significant in determining the catalytic chemistry. Theoretical simulations have been performed to understand the metal/support interactions [1,2]. For example, theorists discovered that electronic and oxygen defects of γ-Al_2O_3 anchor the active particles [1]. Platinum NPs dispersed on γ-alumina is one of the most widely used heterogeneous catalysts and Pt performs extremely well as a catalyst for the oxygen-reduction reaction used in fuel cell industries. Hence, we chose Pt/γ-Al_2O_3 as a model heterogeneous catalyst system to investigate the metal NPs/support interface by electron microscopy methods with the ultimate goal of bridging the gap with theoretical simulations of the interfacial atomic and electronic structure. However, theoretical simulations assume single crystal, planar supports with no impurities, but commercial γ-Al_2O_3 is polycrystalline and irregular in shape [3]. Hence, we are producing a model catalyst support via oxidation of single crystal NiAl to create crystalline and planar γ-Al_2O_3.

We oxidized NiAl(110) from 823K – 1223K at oxygen partial pressures of 10^{-7} - 0.2 atm for 1 to 2 hrs within a controlled atmosphere furnace in order to discover the optimal oxidation conditions forming single crystal γ-Al_2O_3 with flat surface. The microstructure and crystallinity of the oxide films were characterized by X-ray diffraction (XRD) with 2Theta scan, Seemann-Bohlin scan and cross-section transmission electron microscopy (XTEM), Fig.1, 2. The created γ-Al_2O_3 single crystal film follows a Stranski-Krastanov (SK) epitaxial growth, where NiAl(011)[110]||γ-Al_2O_3(111)[211]. This relative orientation matches the Nishiyama-Wasserman (NW) orientation to accommodate 3% lattice mismatch at the NiAl||γ-Al_2O_3 interface, Fig.2.

To deposit Pt NPs on the γ-Al_2O_3 film, Pt was e-beam evaporated onto the γ-Al_2O_3 (111); the particle size ranged from 2-4 nm. XTEM samples were made by using a dual-beam focused ion beam and Fischione Nanomill™, and the interface was characterized by high resolution TEM (HREM), Fig.3. Pt NPs were faceted with an epitaxial relationship of Pt(111)||γ-Al_2O_3 (111). The small Pt particles with less than 0.6 height to width ratio showed a 7% d(111) expansion parallel to the interface suggested strong support effects on the structure of NPs. In summary, γ-Al_2O_3 single crystal film has been prepared successfully via oxidation of NiAl; we will characterize the Pt/γ-Al_2O_3 interface by HREM and EELS and discuss these implications in relation to theoretical predictions [4].

References

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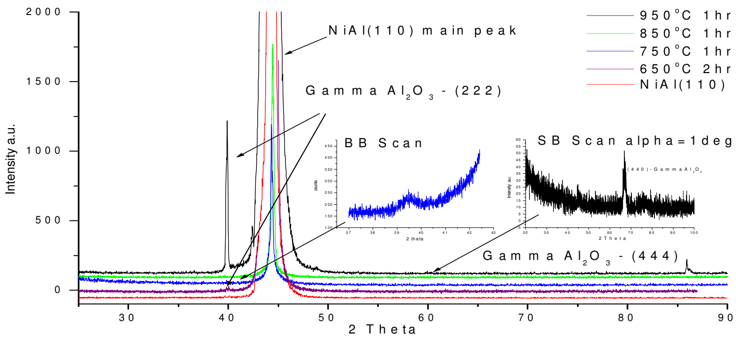

Fig.1 X-ray Diffraction scan of γ-Al_2O_3 /NiAl(110) at various oxidation temperature, (222) γ-Al_2O_3 thin film on the suface; Seemann-Bohlin (alpha=1deg) scan confirm (111) orientation single crystal.

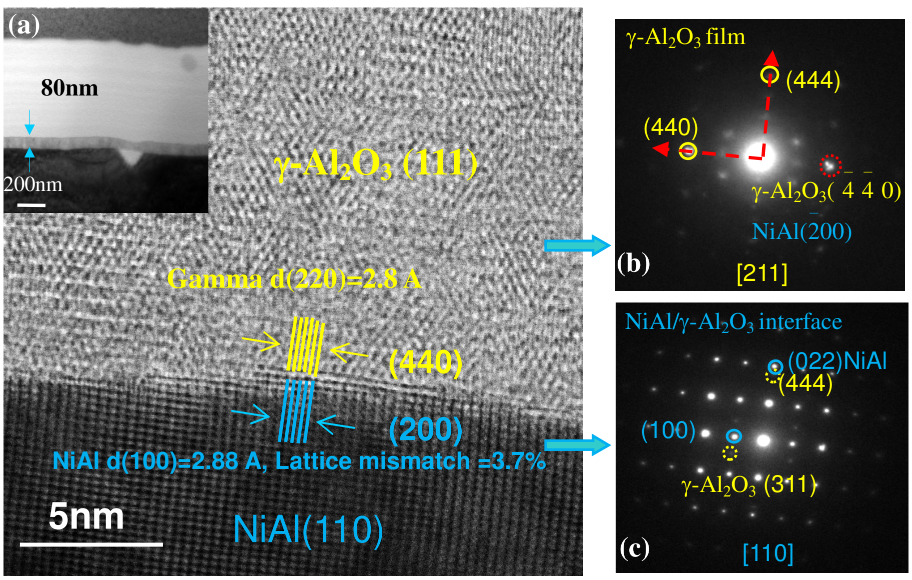

Fig.2 Cross-section HREM image of 850℃ 1hr oxidized NiAl/Al_2O_3 interface, (a). Uniform γ-Al_2O_3 film grows on NiAl substrate. (b). SAD of γ-Al_2O_3 film, (c). Select area diffraction along interface.

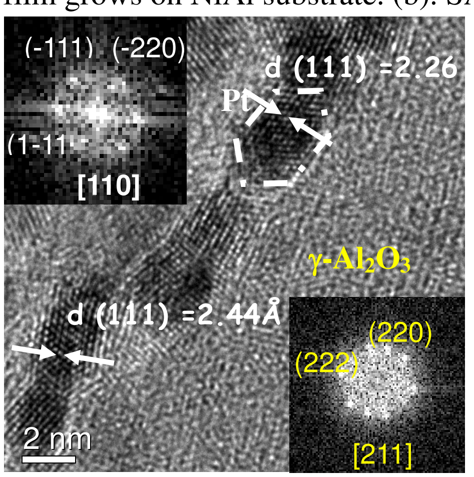

Fig.3 Cross-section HREM image of Pt NPs deposited on γ-Al_2O_3(111) support

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