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EBSD and TEM Microstructural Studies of New Fuel Cladding in Generation IV Sodium-cooled Fast Nuclear Reactors

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A proposed design for Generation IV nuclear systems is a sodium-cooled fast reactor (SFR) [1]. The use of liquid sodium as a coolant requires a high-performance fuel cladding material. One candidate is vanadium alloy V_4Cr_4Ti [2]; however, because of vanadium's high affinity for oxygen, an oxygen concentration as low as 1 ppm in Na [2] provokes vanadium oxide formation, which results in a failure of the fuel cladding protection. To avoid oxygen embrittlement of fuel rods, a diffusion barrier based on a multilayered silicide coating type Si_xV_y was proposed [3]. The combination of electron backscatter diffraction (EBSD) and high resolution transmission electron microscopy (HRTEM) provide full-scale structural characterization and an understanding of the mechanical properties, degradation mechanisms, and substrate adhesion (in the case of protective coatings) of such functional materials.

EBSD and HRTEM techniques are very challenging when investigating coating layers. The ability to prepare a cross-section sample of a substrate with a coating without damaging the coating is a critical to the development of next-generation protective layer. The primary difficulty in cross-section preparation of coatings is keeping the coating on the substrate, which enables the structural characterization, as well as the interface between the coating and substrate without introduction any structural changes and plastic strain [4]. In the case of HRTEM specimen preparation, one of the most popular techniques is gallium focus ion beam (FIB) lift-out. However, a FIB's high-energy Ga ions can damage a sample's crystalline structure by introducing lattice defects (strain induction). FIB milling can also implant gallium ions into the sample surface and can cause surface amorphization. Therefore, an accurate and reproducible sample preparation technique that does not damage the coating or substrate, nor introduce defects or implantation, is required.

In this work, a silicide compound (Si_xV_y) was deposited on a V_4Cr_4Ti vanadium alloy substrate. One sample was cycled in a furnace at 650 °C, the second sample was cycled at 1100 °C. EBSD technique was employed to identify the phases in the multi-layered coating (Figure 1) and to determine growth texture for each phase (Figure 2). An interesting crystallography orientation relationship between the Si_3V_5 / Si_5V_6 and SiV_2 / Si_5V_6 phases: {001} Si_3V_5 // {001} Si_5V_6 and {11-20} SiV_2 // {001} Si_5V_6 was also discovered. HR-TEM study will be performed to complete the structural characterization at the interphase level. This work will illustrate how the use of low-energy argon ion milling can improve and facilitate sample preparation for EBSD and TEM observation of coatings and interfaces.

References

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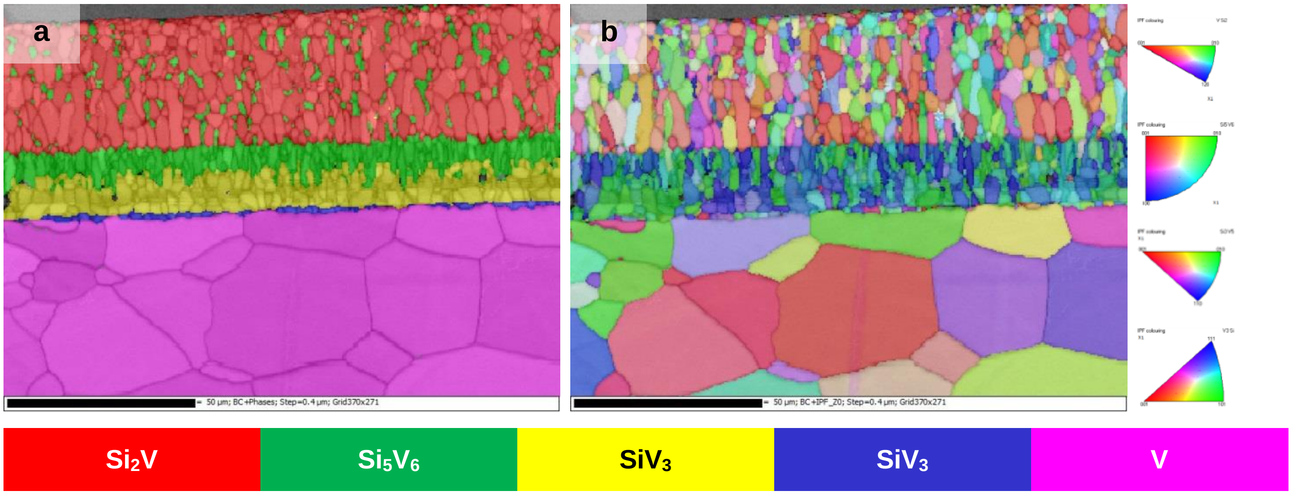

Figure 1. From Si_xV_y samples, a) EBSD phase distribution map, b) IPF color-coded map in layer growth direction after 430 cycles (1 hour per cycle) at 1100 °C.

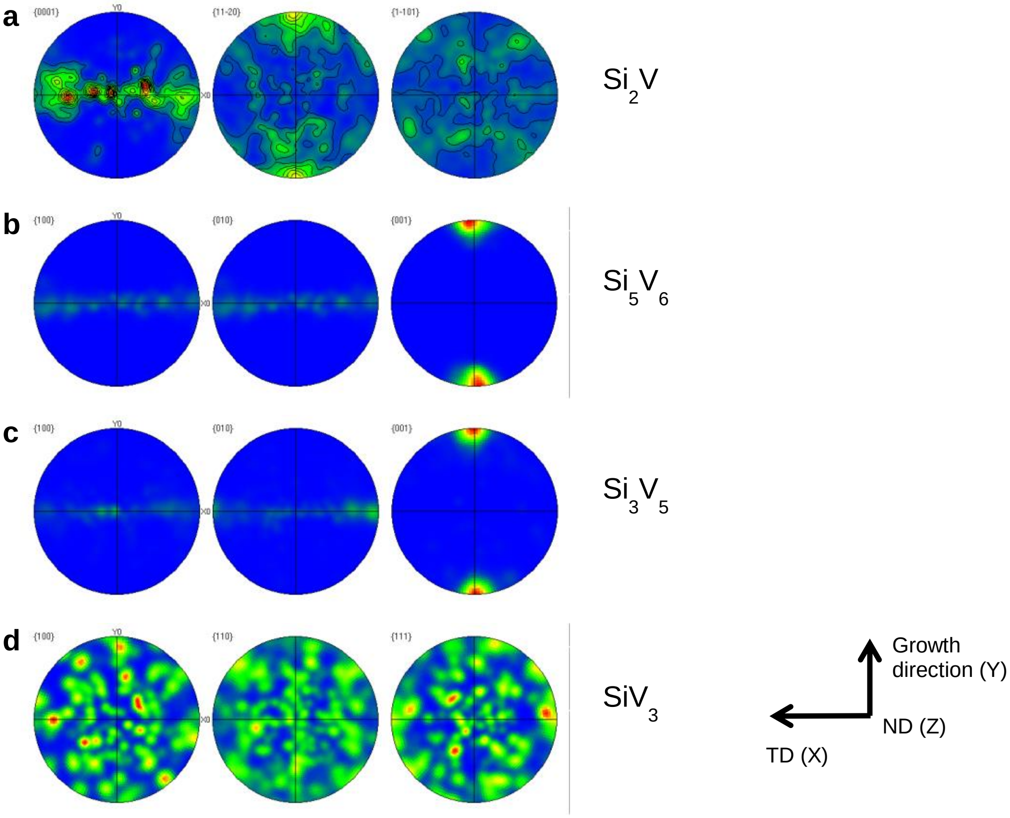

Figure 2. Set of pole figures from the data shown in Figure 1 for Si_xV_y sample cycled at 1100°C: a) Si_2V phase with {0001} and {11-20} fiber texture; b) Si_5V_6 phase with {001} fiber texture; c) Si_3V_5 phase with {001} fiber texture; and d) SiV_3 with random texture.