Phase and Microstructural Characterization using Advanced Techniques: in-situ SEM and TEM-PED

Partha Ghosal
Defence Metallurgical Research Lab.
Kanchanbagh, Hyderabad-500058, India <dr.parthaghosal@gmail.com>

ABSTRACT: Continuous advances in electron microscopy has led to the development of new advanced techniques which have enabled us to get new information about the microstructure which was not available to us before thereby enabling us to have a much more comprehensive understanding of the material at the microstructural level. And, this understanding is the key to manipulate the material properties according to the functional requirement.

This study attempts to highlight the use of two such new /advanced techniques; i) Precision Electron diffraction in TEM. This is a new technique to image the sample in complete zone axis in diffraction mode to get the right orientation of the crystal and match them with the crystal structure diffraction library to get the precise grains orientation in this small volume of the material. ii) In-situ heating / cooling technique in SEM . The in-situ heating cooling technique in SEM allows us to observe the morphological and structural changes as they happen in a material during exposure to varying temperatures. This enables us to have a sound understanding of the material behavior as function of temperature.

These techniques have been used for morphological and structural characterization of high temperature coating materials which include Pt-aluminide (Pt-Al) and Fe-Cr modified silicide and other examples will be discussed, if we have time.

Pt-aluminide (Pt-Al) coatings – They are applied on the Ni-based superalloy components operating in the hot-sections of gas turbine engines. The microstructure of the coating is comprised of the intermetallic B2-NiAl phase. The coating undergoes dynamic phase transformation from B2→ reversible B2/$L1_0$ martensite + γ'-Ni_3Al →γ + γ'-Ni_3Al with thermal exposure. In the present study, we are doing i) in-situ-examination of the effect of temperature on the above phase transformations, ii) identification of local variation of phases/ orientation in the microstructure using PED, & iii) in-situ-examination of the effect of thermal cycling (in high temperature regime) on the structural stability of the coating substrate system.

Fe-Cr modified silicide coatings – They are applied on Nb-base alloys such as C103 for providing oxidation protection at high temperatures. Extensive studies have been done on their ability to withstand high temperature in aerospace applications. However, their

behaviour in low temperature regime (RT to sub zero) is equally important. The present study involves in-situ - examination of the effect of cryogenic temperature on the microstructure and phase stability of the silicide coating using the above mentioned techniques.