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TEMPERATURE-INDUCED STRUCTURAL CHANGES IN PVD ZR-MO-SI-B FILMS: IN-SITU HRTEM INVESTIGATION *

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Currently, much attention is being paid to the development of ZrB_2-based coatings for high-temperature applications. ZrB_2 coatings are rapidly oxidized at T >1100 ° C, therefore, to increase the oxidation resistance as well as strength and durability of ZrB_2, SiC, $TaSi_2$, $MoSi_2$, etc. are alloyed. A comparative study of alloyed coatings has shown that the addition of $MoSi_2$ increases the oxidation resistance of coatings by ~3 times compared with SiC and $TaSi_2$ additives. This work is devoted to a comprehensive study of Zr-Mo-Si-B coatings, including in-situ transmission electron microscopy (TEM) studies of the structure during heating up to 1000°C.

The coatings were deposited by magnetron sputtering in an Ar medium using ZrB_2-$MoSi_2$-MoB ceramic SHS target. The lamellae for TEM were manufactured by ion beam etching using FIB and PIPS II systems. In situ TEM studies of structural-phase transformations were carried out during step heating of lamellae in JEM-2100 microscope. The data obtained were analyzed in comparison with the results of the investigation of the coatings in the as-deposited state and after vacuum annealing at 200-1000°C. The comprehensive study also included the determination of the structure, phase composition, roughness, morphology of coatings by XPS, XRD, FTIR, SEM, and GDOES methods and mechanical properties by nanoindentation.

The results of the TEM show that the column structure of Zr-Mo-Si-B coatings was preserved up to 1000°C. Each columnar grain consisted of equiaxial subgrains. With an increase in temperature, subgrain growth occurred inside the columnar elements. It is important to note that the electronograms recorded at different temperatures are identical and indicate the presence of the h-ZrB_2 phase. The formation of additional crystalline phases was not detected. At the same time, it was revealed that heating led to a decrease in the interplane distance, probably due to the fact that the molybdenum and silicon atoms, leaving the h-ZrB_2 lattice, formed an amorphous $MoSi_x$ phase. Such a change in the composition of the main phase affects strongly on mechanical properties of the coating. At room temperature, the hardness was 39 GPa. Heating up to 200C and 400°C led to a drop in hardness, which can be associated with relaxation of internal stresses during heating. Hardness increased in the temperature diapason of 400-800°C. With and reaching a maximum of 38 GPa, apparently, occurred as a result of the redistribution of molybdenum and silicon atoms between the crystalline phase based on ZrB_2 and the amorphous phase. A further decrease in hardness in the 800-1000 °C area is probably due to an increase in grain size. It was also found that the developed coatings have high thermal stability of the structure and oxidation resistance at temperatures >1200°C.

REFERENCES

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