Investigation of nanoparticles in ODS Ni-free austenitic steel

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Oxide dispersion strengthened (ODS) austenitic steel is a unique material, which has potential for application for future fusion power plants. Introduction of yttrium oxides (Y2O3) to the material improve irradiation resistance and high temperature strength. The nano ?particles play important role in strengthening material by pinning mobile dislocation and stabilization grain boundaries. The interfaces between the matrix and the particles act as sinks for the irradiation-induced defect. Most commercial austenitic steel contain molybdenum and very high amount of nickel which are high-activation elements. It effects in creation of undesirable, radioactive waste. Hence, we have proposed a new material nitrogen containing nickel-free ODS austenitic steel. It is considered as a material for advanced reactor technologies

The Fe-13Cr-20Mn-0.35 Y2O3 and Fe-13Cr-20Mn-0.35 Y2O3-xTi alloys was manufactured by mechanical alloying followed by spark plasma sintering. The as-milled powder was sintered in 950 C or 1000 C for 5 or 15 min. To study the thermal stability of nanoparticles, we have annealed one of the samples (sample sintred in 1000 C for 5) for 1h in temperatures range form 700 C to 1000 C.

We carried out small-angle neutron scattering (SANS) using QUOKKA on assistance of ISSP. We used neutrons with the wavelength 2.95 Å and q-range from 6 x 10^-4 A^-1 to 0.7 A^-1 with standard and focusing lens optics The measurements were held in the ambient temperature with non-polarized neutron. Source aperture to sample aperture distance was 3, 12 and 20 m and focusing lens optics (L1=12m L2=1p3m offset_apx5). The measured data was corrected by using the results of the glassy carbon and thickness and analyzed by Irena software using Size Distribution. To obtain information about size, number and chemcial composition of nanoparticles, we have applied the alloy contrast variation analysis (ACV), combining USAXS and SANS data. The ACV method is based on the difference of X-ray and neutron scattering length of each element, which is described as a ratio of intensity between SANS and SAXS.

In low-q regoin, we observed clear humps in all samples. Increase of sintering temperature causes humps shifting to lower q-value. Samples sintered in 950 C have a mean radius around 200 A. In 1000 C occurred particles growth up to 270 A for samples sintered in 5 min and 380 for the samples sintered for 15 minutes. The biggest nanoparticles were obtained in samples with 0.1% Ti sintered in 950 C for 15 min.

Next, we have considered the SANS and USAXS curves in the ACV analysis. The analysis indicates presence of two population of Y2O3, ε-martensite and MnO nanoparticles, what is correct according to XRD, TEM and EDS results. The presence of very fine Y2O3, radius round with 20 A is suggested, it has occurred reprecipitation of dissolved Y2O3 during mechanical alloying. The results of ACV analysis of annealed samples show increase of volume distribution of the finest Y2O3 after annealing in 800 C. The nanoparticles are stable up to 900 C. In 1000 C growth of all nanoparticles occurred, and the third population of coarse Y2O3 appeared.