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# Spray of Mechanically Alloyed F/M-ODS Steel Powder

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## 1. Introduction

Oxide dispersion strengthened (ODS) steels have drawn wide attention as a promising candidate in-core structural materials of the future advanced fast reactor. ODS steels have excellent resistance to creep and irradiation induced swelling [1,2]. It is well known that uniform nano-oxide dispersoids act as pinning points to obstruct dislocation and grain boundary motion in ODS steel. They would have the ability to control irradiation damage, as the resultant gas bubbles are formed around the dispersed oxide particles rather than at grain boundaries. In KAERI, particular attention has been paid to the fabrication of thin-walled cladding tubes of ODS steels, because tubing process of ODS is somewhat difficult. The cladding tube of a sodium fast reactor (SFR) is dimensioned to be 7.4 mm in outer diameter, 0.54 mm in wall thickness and 2500 mm in length. Ordinary processes for ODS tubing is described below. First, mechanically alloyed powders were charged in a steel capsule, and degassed at 500℃ for 1 hour. After a pre-heating in the furnace at 1100℃ for 2 hours, the capsules were extruded with a 6.4:1 extrusion ratio. Next, ODS steels were heat-treated to show a sufficient hardness level for the tubing including pilgering and the cold-drawing process. Tens of processes, pilgering, drawing and intermediate heat treatments should be followed. As described above, so many complicated processes are involved for ODS tube manufacture. The ODS materials are inevitably brittle, because they are consolidated from powders. Sometimes, they cannot endure the severe shear deformation; the material can be easily broken during tubing. In this study, the thermal/cold spray was considered to improve the productivity of ODS steels. In recent years, the development of the high velocity oxygen fuel (HVOF) process has enabled the wide usage of carbide and metal-based coatings for demanding applications [3,4]. In HVOF processes, kinetic energy of particles is more important than temperature. These processes use high-impact energy to produce dense coatings at relatively low temperatures. HVOF is advantageous for coating large components, produce high-density freeform without degeneration due to high-temperature effects. Similar with HVOF, Cold spray produces deposits that are oxide-free and fully dense with acceptable mechanical properties. First, the coatings can exhibit wrought-like microstructures with near theoretical density values; second, the spray trace is small and well defined allowing for precise control on the area of deposition; last, the coatings can be produced with compressive stresses, thus ultra thick (5-50 mm) coatings can be built-up without adhesion failure [5,6].

## 2. Methods and Results

The material used in this study was a F/M ODS steel (Fe(bal.)-10Cr-1Mo-0.5Mn-0.1V-0.25Ti-0.35Y2O3 in wt.%). HVOF machine named diamond jet (DJ-2600) was used to spray the ODS powder, feeding rate was 35-65 g/min, carrier gas was nitrogen, source to objective distance (SOD) was 200~300 mm, and traverse speed was 500 mm/s. A cold spray machine was also used, the temperature was 800℃, pressure was 35 bar, SOD was 32 mm, and traverse speed was 50 mm/s. The spray processed specimens were observed by SEM and TEM microscopy, the size of coating layer, porosity, the identification of oxide particle were conducted. The heat-treatment of ODS powder was performed using quartz tube, the heat treatment temperature was 600℃, 800℃, 1000℃, and 1200℃, respectively. Powders were gathered using magnet, consolidated by glue and epoxy. Electro-polishing were performed on the mechanically polished surface, micro-hardness was measured.

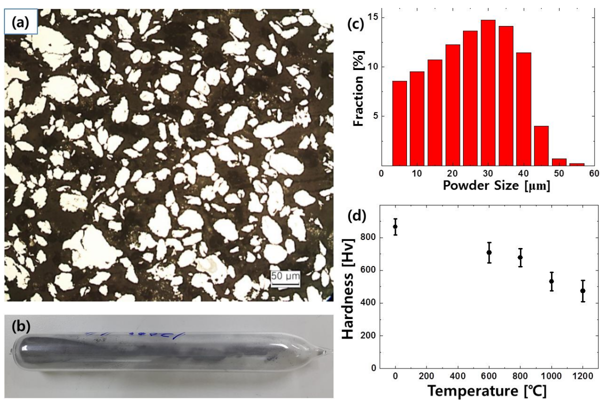


Fig. 1. (a) Mechanically Alloyed powder of ODS for the spray process. (b) Quartz tube for heat-treatment of powders. (c) Size distribution of mechanically alloyed

powders. (d) The heat-treatment temperature and corresponding hardness variations were shown.

In Fig. 1(a). mechanically Alloyed powder of ODS for the spray process is shown. The average diameter was estimated by 30 μm, lots of them consist of flake type powders. Quartz tube for heat-treatment of powders are shown in Fig. 1(b). specimens were sealed in vaccum for preventing oxidation. The size distribution of mechanically alloyed powders are shown in Fig. 1(c). The range of sizes stretch from 5 to 50 μm. The heat-treatment temperature and corresponding hardness variations were shown in Fig. 1(d), the hardness of powder was reduced almost half after 1200℃ heat-treatment. The 12 specimens of HVOF process were compared in Fig. 2. The optical micrograph from the plane view is shown in Fig. 2(a), the surface of the coating looked homogeneous. The scanning electron micrograph from the cross section view are shown in Fig. 2(b), the density and thickness of layers were different from conditions (source to objective distance, flow rate of powders). Adequate process condition was found in the specimen number 2, 5 and 10. In Fig. 3, TEM micrographs of HVOF processed ODS are shown. Heat affected zone in the coated ODS layer was displayed by dark contoured boundaries. Relatively high number density of $Cr_2O_3$ phases were observed Fig. 3(b) in the spacing among powders. They can be the critical defects for the delamination of the film. Inside the powders, small nano oxide particles were observed as shown in Fig 3(c), the diameter was rougly 10 nm. The phase was identified as $Y_2Ti_2O_7$.

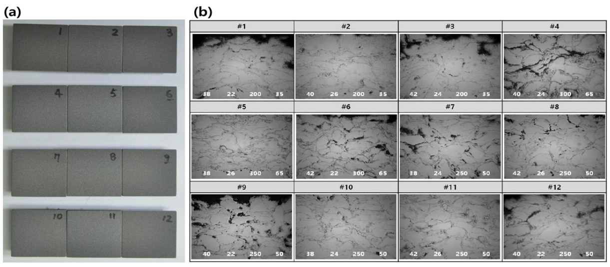

Fig. 2. Images of HVOF processed ODS layer. (a) optical micrograph from the plane view. (b) scanning electron micrograph from the cross section view

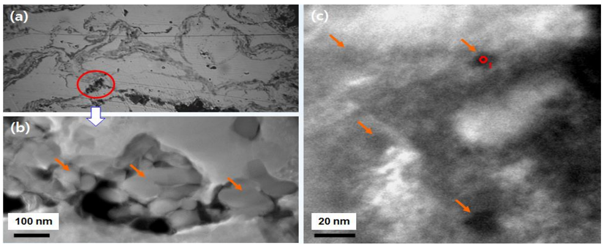


Fig. 3. (a) Heat affected zone in the coated ODS layer (b) $Cr_2O_3$ phases (c) $Y_2Ti_2O_7$ nano particles.

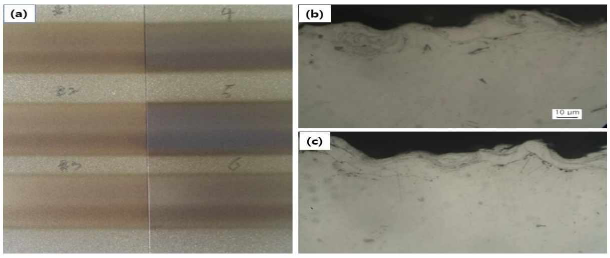


Fig. 4. cold spray processed ODS layer. (a) optical micrograph from the plane view. (b) scanning electron micrograph from the cross section view.

The cold spray processed ODS layer is shown in Fig. 4. The optical micrograph from the plane view and the scanning electron micrograph from the cross section view are shown in Fig. 4(a) and 4(b), respectively. The thickness of layer was only ~10 μm, island type. The hardness of mechanically alloyed powder is too high, the amounts of work hardening during mechanical alloying have to be reduced by pre-heating process before cold spray. Modification of process variable and heat treatment methods are under consideration. The thermal annealing of ODS powder without agglomeration is the key factor to jump over the current obstacle.

## 3. Conclusions

In this study, thermal/cold spray of mechanically alloyed F/M-ODS steel powder was investigated. HVOF was applied as one of the thermal spray method and it was very effective to produce thick film. However, the film is filled with undesirable Cr oxides in the spacing among powders. It is expected that the cold spray is more desirable to obtain an oxide-free film, unfortunately, cold spray was not applicable with current powder status. The hardness of mechanically alloyed powder was too hard to form a dense film. The modified cold spray tests are steel on going.

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