# Fabrication and characterization of aluminum-containing ferritic ODS alloys for improved corrosion resistance

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**Abstract.** Future generation nuclear reactors will have demanding requirements on materials. Present materials are unable to cope with the desired operating temperatures and environments. Ferritic ODS alloys are candidate materials which might meet these requirements. Especially, alloys with additional aluminum content have a large potential to withstand the corrosive environments. In the present study, 12-14%Cr ferritic ODS alloys with varying aluminium content produced by mechanical alloying are investigated. After mechanical alloying of a pre-alloyed steel powder with aluminum in an attritor ball mill, the process was investigated by SEM and TEM analysis of the milled powder particles. After compacting by hot isostatic pressing and hot-rolling followed by annealing, the mechanical properties of the materials were assessed by tensile tests and hardness measurements. The results showed that the aluminum content has a big influence on the powder particle size during mechanical alloying. Also a drop in the yield strength could be observed. All further results are analyzed, compared, and discussed in this paper.

## 1. Introduction

Oxide dispersion strengthened ferritic steels, which have been developed in the recent years have superior properties when compared to conventional high-chromium ferritic steels. These properties qualify them as candidate materials for future generation nuclear power plants. Alloys containing between 12 and 14 % chromium with an addition of 2-4% of aluminium have a large potential to withstand high corrosive environment such as supercritical water reactors and liquid metal cooled conditions.

## 2. Materials and processes

In this present study, the production of four different ODS alloys was done by mechanical alloying (MA) pre-alloyed powders in an attritor ball mill (ZOZ Simoloyer CM02) for 80 hours at 1200 rpm. The basic alloy powder has a composition of Fe-13Cr-1W-0.3Ti and was produced by argon-gas-atomization by Nanoval, Berlin. 0.3 wt.% Yttrium were added in the form of a powder of an intermetallic phase of $Fe_2Y$, also produced by Nanoval. The variation of the aluminium content was done by adding different amounts of $FeAl_3$ powder before mechanical alloying. The dilution of other alloying elements of the pre-alloyed powder by the addition of the iron intermetallic powders was compensated by adding Chromium (Cr) and Titanium-Hydrate ($TiH_2$) powders to the high-alumium containing alloys (2,3 and 4% Al).

All processing after MA was done in argon inert gas atmosphere without exposing the powders to air. Following, the material was put into steel cans and HIPped for 2 h at 1150°C with an applied pressure of 100 MPa. After HIPing, the cans were hot-rolled (HR) at 1100°C from a diameter of 40 mm to 6 mm thickness in 5 passes with reheating after each pass at TU Clausthal.

Table 1. Chemical composition of the produced alloys

| No. | Cr | W | Ti | Y | Al | Fe |
|---|---|---|---|---|---|---|
| 0 (ref.) | 13 | 1 | 0.3 | 0.3 | - | bal. |
| 2 | 13 | 1 | 0.3 | 0.3 | 2 | bal. |
| 3 | 13 | 1 | 0.3 | 0.3 | 3 | bal. |
| 4 | 13 | 1 | 0.3 | 0.3 | 4 | bal. |

Powder particle sized distribution measurements were done on a Horriba LA-950 laser scattering particle analyzer which was operated in flowing iso-proponal condition. All measurements were carried out multiple times at averaged over all results. TEM specimens were produced by thin film cutting with microtomy. The full process was described elsewhere[1] .
Miniaturized round specimens with 7.6 mm x 2 mm gauge length were used for the tensile tests, which were performed at temperatures ranging between 23°C and 700°C using a strain rate of $1{,}6 * 10^{-6} ms^{-1}$ All specimens were cut by electro-discharge-machining (EDM) and taken out in L-T orientation in the (elongated) rolling-direction. Vickers hardness tests a universal hardness tester with 30 kp loading and averaged over 3 measurements per specimen.

## 3. Experimental results and discussion

The particle size distribution of the mechanically alloyed powders showed a big variation with increasing alumium content. A major increase in the particle size can be observed after the addition 3 and 4 wt.% of aluminium . Slight coarsening of the powder particles can be observed with the 2% al alloy (Figure 5). The increase of the powder particle size and coarsening of the particles with increasing al-content is caused by the softening of the material due to the al-content. During the mechanical alloying, softer materials tend to form larger particles after milling. The MA-process is characterized by two dominant mechanisms. Welding of powder particles due to the high milling energies leads to coarser particles at the beginning of the process. When the damage and deformation in the powder reaches a certain level, fracture of particles becomes the dominant mechanism [2]. This was shown in earlier works on ferritic (non-al) ODS alloys [3]. By looking at the particle size measured after 80 hours milling, it leads to the conclusion that the milling process is still dominated by the cold welding process, especially when compared to the reference (non-Al) alloy.

**Table 1 mean particle sizes of different alloys**

| Alloy | 0%-Al | 2%-Al | 3%-Al | 4%-Al |
|---|---|---|---|---|
| mean particle size ($d_{50}$) [µm] | 40.81 | 42.52 | 81.13 | 109.71 |

The examinations by transmission electron microscopy were performed in high angle annular dark field mode (HAADF) combined with energy dispersive x-ray spectroscopy (EDS). The obtained images and elemental mappings can be found in Figure 1and Figure 2. The results of the 2%-Al alloys (Figure 1) show larger areas of chromium enrichements and very small clusters with higher alumium concentration. This phenomen increases when looking at the the 3%-Al materials (Figure 2). These

enrichements are an effect of the mechanical alloying can be neglected for the properties and structur of the final materials. By looking at the Fe-Al and Fe-Cr phase diagramms, it becomes obvious that both chromium and aluminium will form a solid solution with the iron matrix during compacting and thermo-mechancal treatments due to the high temperatures and slow cooling rates.

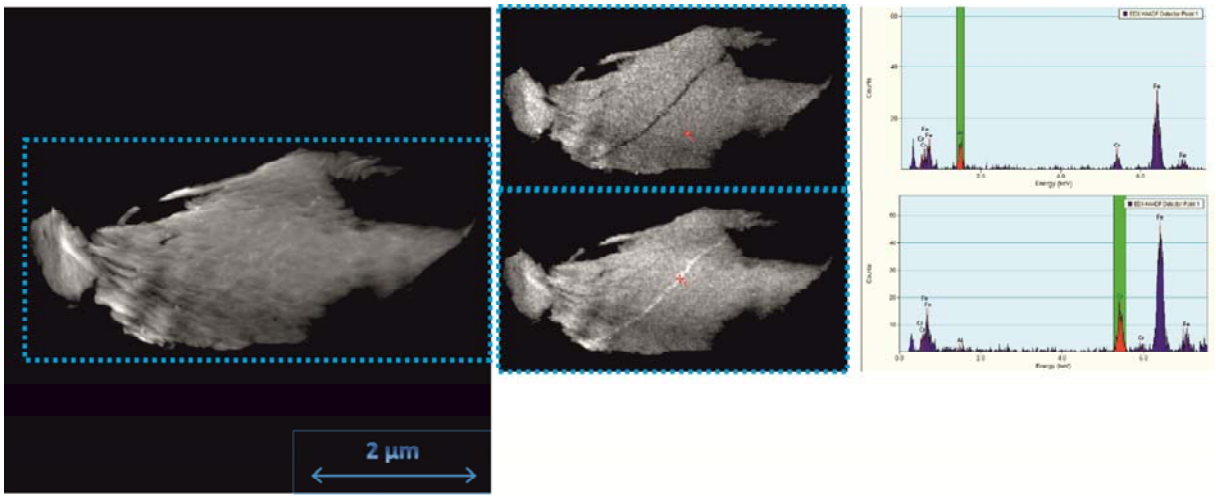


**Figure 1 HAADF image and EDS map of 2% al alloy**

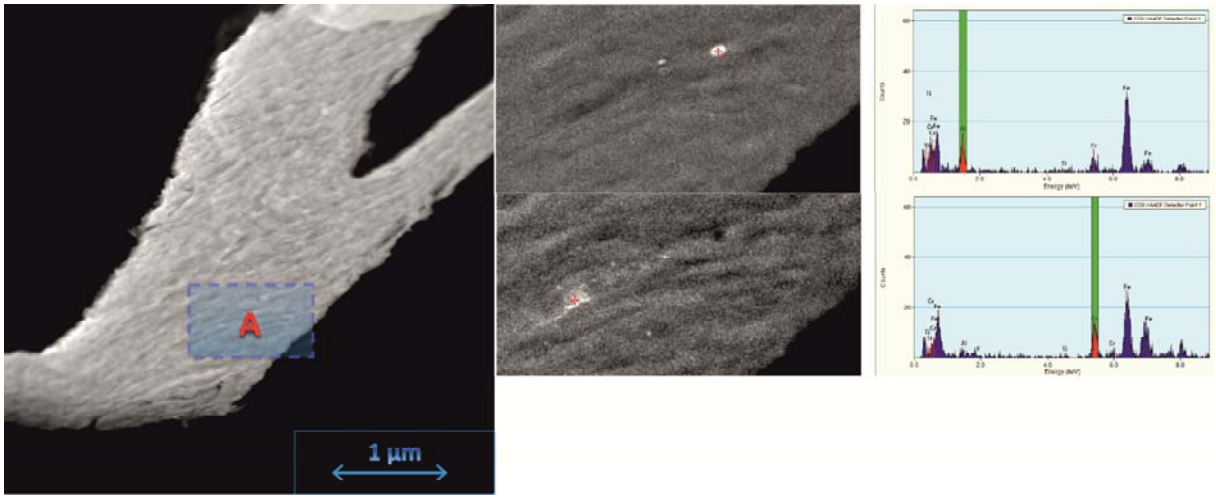


**Figure 2 HAADF image and EDS elemental map of 3% Al alloy**

The Vickers hardness of the alloys after HIPping and hot rolling shows a significantly lower hardness of the al-containing materials (Figure 4). In the mechanical properties, a lower strength can also be observed. The addition of alumium causes the yield strength to drop more than 300 MPa, when compared to the reference alloy (Figure 3). A drop in (yield) strength of the material was expected and has also been seen in works by other groups [4]. A difference in the density and size of nano-oxide clusters inside the microstructure causes the weaker mechanical properties. TEM characterization are planned for the future to reveal this matter. However, it is remarkable that the strength at the desired operating temperatures (550°C+) is independent of the alumium content.

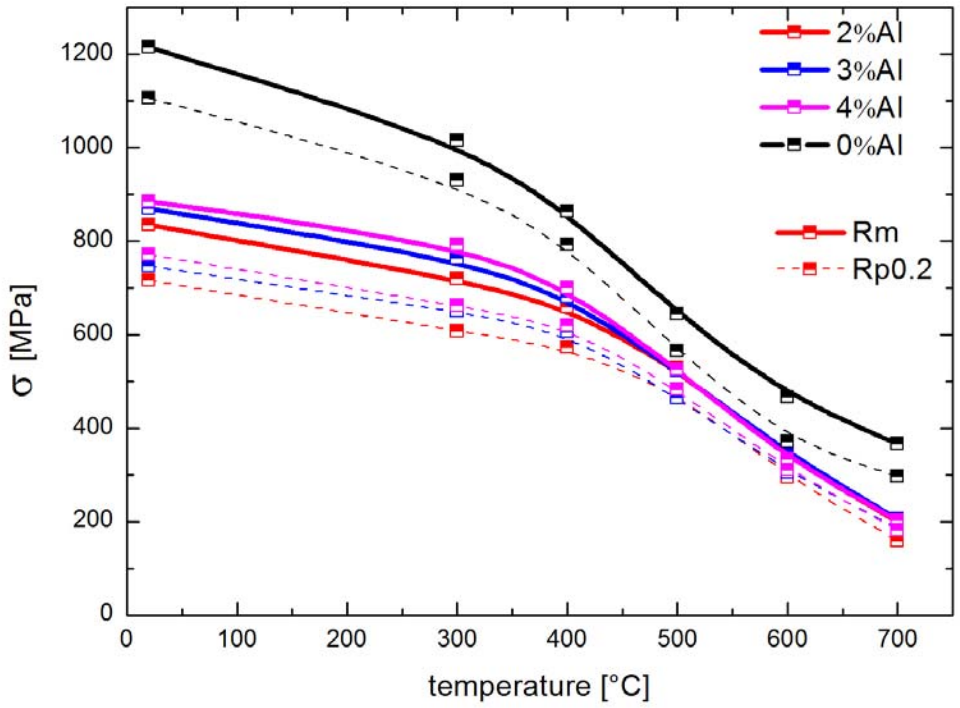


**Figure 3 tensile testing of alloys (yield strength and ultimate tensile strength)**

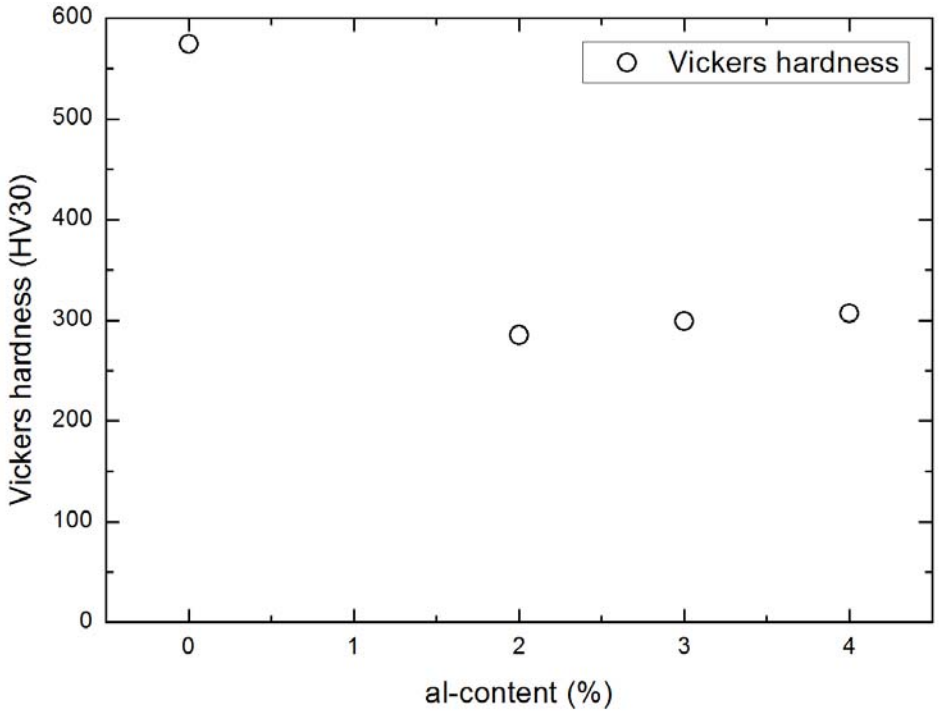


**Figure 4 Vickers hardness of alloys**

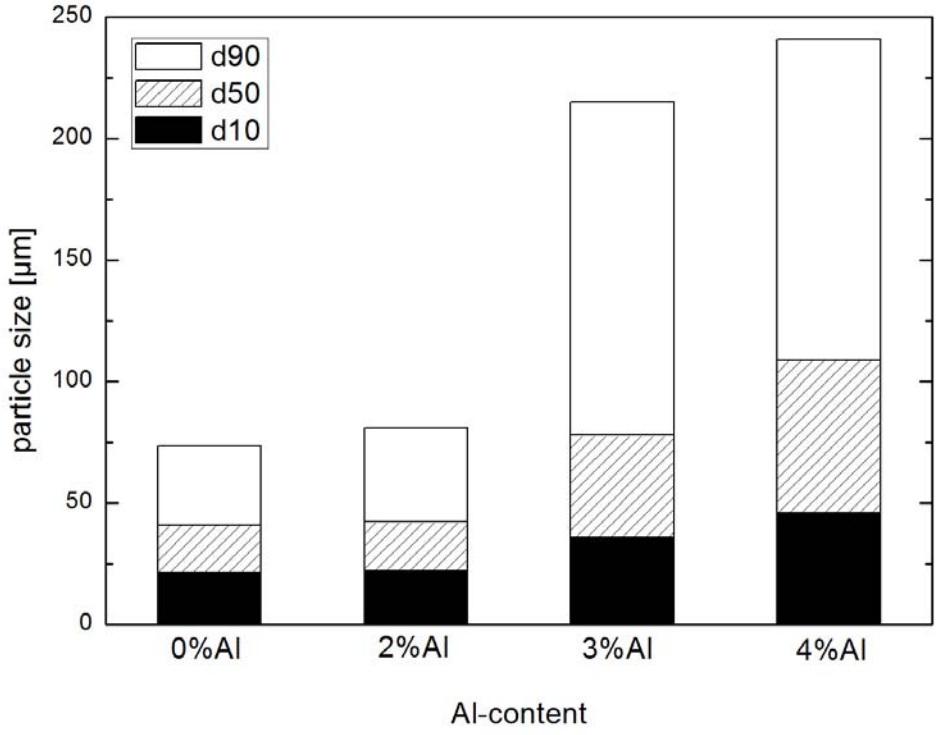


**Figure 5 powder particle size distribution**

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