

## Tensile Strength of Surface-treated Zircaloy-4 with $Y_2O_3$ Particles using a Laser Beam

Yang-Il Jung,\* Jung-Hwan Park, Dong-Jun Park, Young-Ho Lee, Byoung-Kwon Choi, Hyun-Gil Kim, Jae-Ho Yang  
Korea Atomic Energy Research Institute, 989-111 Daedeok-daero, Yuseong, Daejeon, 34057, Republic of Korea

\*Corresponding author: [yijung@kaeri.re.kr](mailto:yijung@kaeri.re.kr)

### 1. Introduction

Accident tolerant fuel (ATF) cladding is being developed globally after the Fukushima accident with the demands for the nuclear fuel having higher safety at normal operation conditions as well as even in a severe accident conditions. Korea Atomic Energy Research Institute (KAERI) is one of the leading organizations for developing ATF claddings [1,2]. One concept is to form an oxidation-resistant layer on Zr cladding surface. The other is to increase high-temperature mechanical strength of Zr tube.

The oxide dispersion strengthened (ODS) zirconium was proposed to increase the strength of the Zr-based alloy up to high temperatures [2-5]. The ODS treatment on the Zr surface layer was successfully performed using a laser beam scanning (LBS) process, as shown in Fig. 1 [4]. High-power laser beam was exposed on the zirconium surface previously coated by oxides – typically  $Y_2O_3$ . The dispersed oxide layer was formed by the penetration of oxide particles into Zr alloys.

According to our previous investigations [3-5], the tensile strength of Zircaloy-4 was increased by up to 20% with the formation of a thin dispersed oxide layer with a thickness less than 10% of that of the Zircaloy-4 substrate. However, the tensile elongation of the samples decreased drastically. The brittle fracture was a major concern in development of the ODS Zircaloy-4. In this study, the behavior of mechanical strength with respect to the test temperature is investigated.

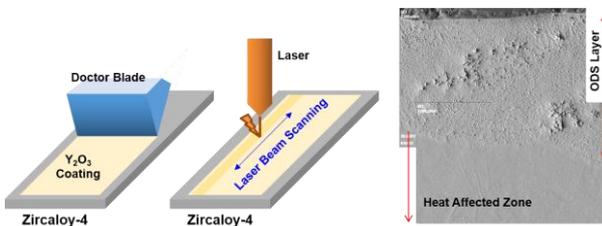


Fig. 1. Schematic illustration of ODS treatment using a laser beam scanning [4].

### 2. Methods and Results

#### 2.1. Fabrication of ODS Zircaloy-4 Plate Samples

A Zircaloy-4 (Zr-1.5Sn-0.2Fe-0.1Cr) alloy sheet with 2 mm in thickness was used as a substrate. Zircaloy-4 plates showed a fully recrystallized microstructure.

Oxide powder of  $Y_2O_3$  (99.9%, 1  $\mu$ m, Alfa Aesar, USA) was purchased, and coated on Zircaloy-4 sheet with the thickness of 10–55  $\mu$ m. Oxide coating was prepared using a water-based slurry containing a polyvinyl alcohol (PVA) as a binder. The slurry was coated on Zircaloy-4 plate by a doctor blade, and dried in an oven at 80°C for 30 min. The coated Zircaloy-4 samples were laser beam scanned by a continuous wave diode laser with a maximum powder of 250 W (PF-1500F, HBL Co, Korea). To prevent oxidation during the LBS, Ar gas was continuously blew onto the melting zone through a laser nozzle. Schematic illustration of LBS was shown in Fig. 1. The wavelength of the emitted laser beam was 1064 nm, and the beam diameter was 230  $\mu$ m. Hatching distance and scan speed were set as 0.4 mm and 10 mm/s, respectively.

#### 2.2. Fabrication of ODS Zircaloy-4 Tube Samples

Zircaloy-4 tubes with an outer diameter of 9.5 mm and a wall thickness of 0.57 mm were used. The as-received Zircaloy-4 tubes showed a cold-worked and stress-relieved microstructure. The tubes were cut in length of 450 mm and cleaned with alcohol and acetone.  $Y_2O_3$  was coated on the cleaned Zircaloy-4 tubes by a dip-coating method. To prepare the dip solution,  $Y_2O_3$  powder was mixed with distilled water containing 10 wt.% PVA. The amount of PVA was 3 wt.% of the  $Y_2O_3$  content. The solution was mixed for 24 h using zirconia balls. Zircaloy-4 tubes were immersed in the solution and then slowly drawn out to form a coating layer. The wet-coated tubes were dried in a vacuum oven at 80°C for 20 min. The  $Y_2O_3$ -coated Zircaloy-4 tubes were scanned by the laser (PF-1500F). The ODS alloy layer was formed at a laser beam power of 210 W and scan speed of 10 mm/s. The laser beam was scanned continuously along the circumferential direction with an overlap distance of 0.4 mm. To prevent oxidation and blow off the PVA binder, Ar gas was continuously blown on the samples' surfaces during laser processing. Cooling water was supplied to the inside of the tubes to release the induced heat.

#### 2.3. Tensile Test at Room Temperature

Fig. 2 shows the stress–strain curves for the ODS plate samples under tensile test at room temperature (RT) [4]. The tensile strength and elongation of fresh Zircaloy-4 were about 510 MPa and 35%, respectively. As a comparison, laser-beam-scanned Zircaloy-4 plates

without a  $Y_2O_3$  coating (LBS only) was tested, and showed increased tensile strength of about 630 MPa. The martensitic phase transformation during laser processing is the origin of the strength increase of the LBS samples. The ODS samples, furthermore, exhibited a much higher tensile strength than fresh and LBS Zircaloy-4 samples. The obtained tensile strength of the ODS samples was 651–695 MPa. However, the elongation decreased drastically to 10–21%. An abnormal drop in the tensile strength was observed at the maximum load for the ODS samples. The brittle surface layer after oxide dispersion strengthening treatment accounts for this behavior.

Fig. 3 shows the stress–strain curves for fresh, laser-beam-scanned (LBS), and surface-treated Zircaloy-4 tube samples during ring tension tests at RT. The tensile stress was calculated by dividing the applied load by the

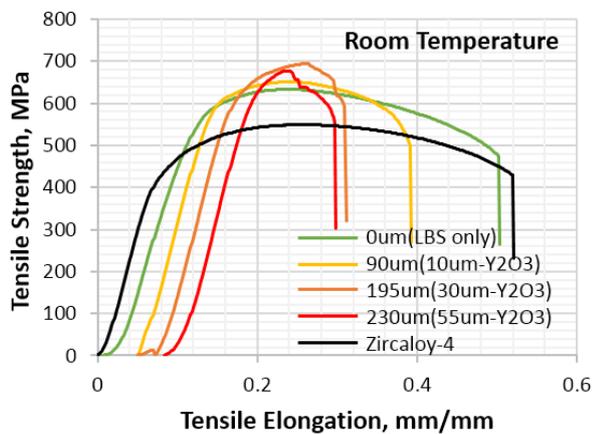


Fig. 2. Stress-strain curves of ODS Zircaloy-4 plate samples tensile tested at RT with respect to the thickness of the ODS layer [4].

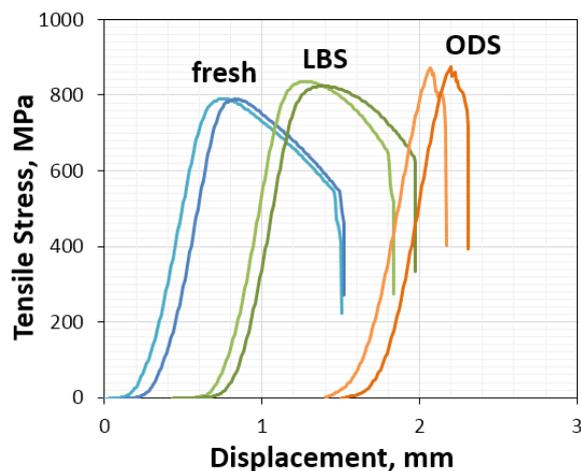


Fig. 3. Stress-strain curves of ODS Zircaloy-4 tube samples tensile tested at RT with respect to the thickness of the ODS layer.

cross-sectional area. The ultimate tensile strength of fresh Zircaloy-4 were about 790 MPa. When Zircaloy-4 was scanned by the laser beam, the tensile strength increased to about 840 MPa. As in the case of plate samples, the martensitic phase transformation during laser processing is the origin of the strength increase of the LBS samples. The ODS samples, furthermore, exhibited a much higher tensile strength than fresh and LBS Zircaloy-4 samples. On the contrary, the tensile elongation decreased dramatically, showing an abrupt drop in the applied tensile load.

#### 2.4. Tensile Test at Elevated Temperatures

The strengthening of Zircaloy-4 by the ODS layer was very effective at elevated temperature [4,5]. Fig. 4 shows the stress–strain curves of the ODS samples tensile tested at 380 °C. The ultimate tensile strength and elongation of fresh Zircaloy-4 were about 210 MPa and 47%, respectively. The ODS samples exhibited a tensile strength of about 355 MPa, which is almost 70% greater than that of fresh Zircaloy-4. In addition, the elongation did not exhibit a stress drop or a dramatic decrease, which had been observed in the test at RT (Fig. 2). The tensile elongation of the ODS samples was 33–37%. The values are about 30% less than that of the fresh Zircaloy-4 sample.

Fig. 5 shows the stress–strain curves for fresh and surface-treated Zircaloy-4 samples ring tensile tested at 380°C and 500°C. The average tensile strength of Zircaloy-4 increased from 500 to 575 MPa at 380°C and from 385 to 468 MPa at 500°C with the formation of the ODS layer. Moreover, the severe decrease in tensile elongation was not observed. This is meaningful for the application of ODS Zircaloy-4 as an in-core nuclear structural material, because the working temperature of fuel cladding tubes is 300–400°C.

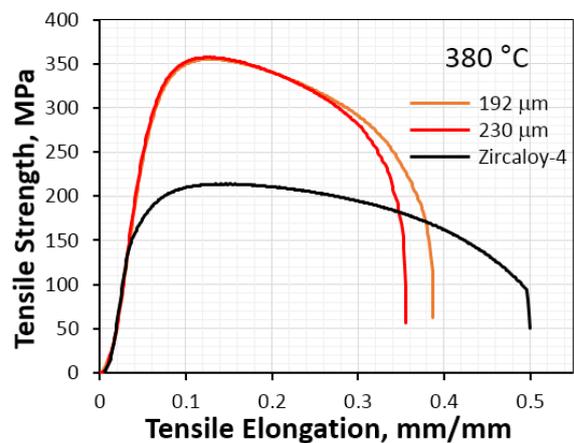


Fig. 3. Stress-strain curves of ODS Zircaloy-4 tube samples tensile tested at 380°C with respect to the thickness of the ODS layer [4].

### 3. Conclusions

Surface treatment was performed by a laser beam to form a dispersed oxide layer in Zircaloy-4. Laser beam scanning of a tube coated with yttrium oxide ( $Y_2O_3$ ) resulted in the formation of a dispersed oxide layer in the tube's surface region.  $Y_2O_3$  particles penetrated the Zircaloy-4 during the laser treatment and were distributed uniformly in the surface region. The oxide dispersion strengthened (ODS) layer increased the mechanical strength of Zircaloy-4. The tensile strength of Zircaloy-4 increased by 10–20% with the formation of the dispersed oxide layer. The strengthening of Zircaloy-4 by the ODS layer was very effective at elevated temperature. The brittle fracture observed at room temperature was changed in ductile at elevated temperatures. The formation of ODS layer is useful for manufacturing Zr alloy tubes with enhanced safety and accident tolerance because of the increased strength up to high temperatures.

### Acknowledgment

This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIP) (No. 2017M2A8A5015058)

### REFERENCES

- [1] Y.-H. Koo, J.-H. Yang, J.-Y. Park, K.-S. Kim, H.-G. Kim, D.-J. Kim, Y.-I. Jung, K.-W. Song, "KAERI's development of LWR accident-tolerant fuel," Nucl. Technol., 186, 295-304 (2014).
- [2] H.-G. Kim, J.-H. Yang, W.-J. Kim, Y.-H. Koo, "Development status of accident-tolerant fuel for light water reactors in Korea," Nucl. Eng. Technol., 48, 1-15 (2016).
- [3] H.-G. Kim, I.-H. Kim, Y.-I. Jung, D.-J. Park, J.-Y. Park, Y.-H. Koo, "Microstructure and mechanical strength of surface ODS treated Zircaloy-4 sheet using laser beam scanning," Nucl. Eng. Technol., 46, 521-528 (2014).
- [4] Y.-I. Jung, H.-G. Kim, I.-H. Kim, S.-H. Kim, J.-H. Park, D.-J. Park, J.-H. Yang, Y.-H. Koo, "Strengthening of Zircaloy-4 using  $Y_2O_3$  particles by a laser-beam-induced surface treatment process," Mater. Des., 116, 325-330, (2017).
- [5] Y.-I. Jung, H.-G. Kim, H.-U. Guim, Y.-S. Lim, J.-H. Park, D.-J. Park, J.-H. Yang, "Surface treatment to form a dispersed  $Y_2O_3$  layer on Zircaloy-4 tubes," Appl. Surf. Sci., in press.

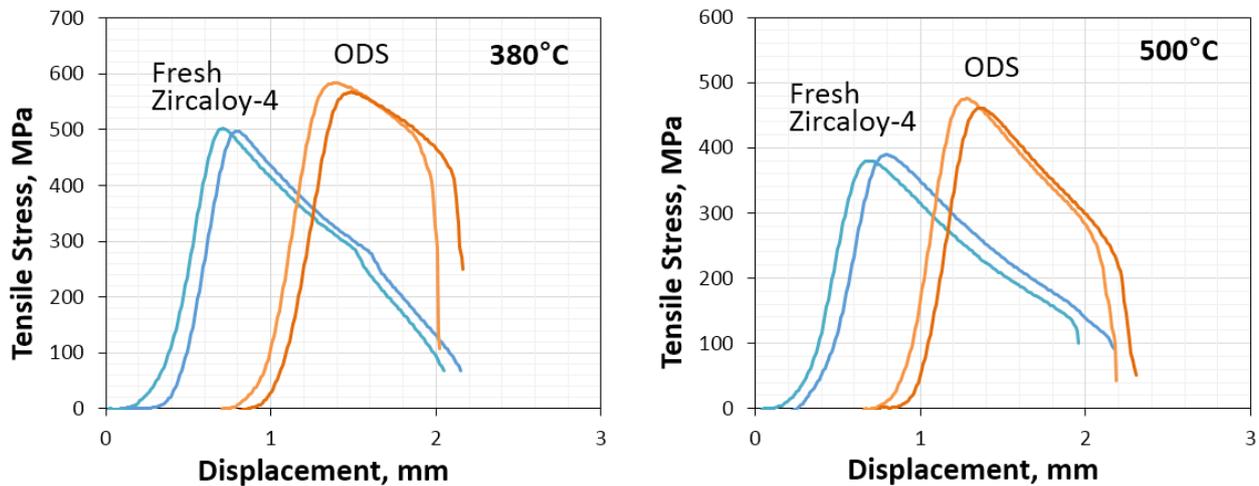


Fig. 4. Tensile stress-strain curves for the ODS Zircaloy-4 tube samples at elevated temperatures of 380°C and 500°C, respectively.