[Corresponding Author]
Name : Hiroyuki Noto
Telephone number: +81-572-58-2314
Fax number : +81-572-58-2254
E-mail address : noto.hiroyuki@nifs.ac.jp

Transformation super plasticity deformation of reduced activation ferritic / martensitic steel

Hiroyuki Noto[1], Yoshimitsu Hishinuma[1], Takeo Muroga[1]
[1]*National Institute for Fusion Science, 322-6 Oroshi, Toki 509-5292, Japan*

Abstract

Reduced Activation Ferritic/Martensitic (RAFM) steel is a promising candidate for the blanket structural materials of fusion reactors. One of the key issues in the use of RAFM for the blanket is its low workability. As a solution of this issue, innovative processing technology based on the transformation super plasticity (TSP) was proposed. In general, TSP is known as high temperature creep deformation which is induced by phase transformation. In this study, tensile tests under constant load were carried out with cyclic temperature variation for a RAFM steel ~~and an oxide dispersion strengthened (ODS) RAFM steel~~ to investigate the effect of cyclic temperature variations on elongation. The result of the tensile test under constant load for the RAFM steel with cyclic temperature variations exhibited a macro-elongation to 172 %. ~~However, the ODS steel did not exhibit the macro-elongation, but showed grain boundary cracking~~. The results of the tensile tests with varying loads showed the possibility of reducing the necessary time and enhancing the controllability for the formation of RAFM steel products using TSP.

Keywords: transformation super plasticity, reduced activation ferritic/martensitic steel, oxide dispersion strengthened steel, creep deformation

1. Introduction

Reduced Activation Ferritic/Martensitic (RAFM) steels, which have been investigated over the decades, were designed to reduce neutron irradiation induced activation by replacement of some minor elements (molybdenum, niobium) of the conventional heat-resistant steels (ex. 9Cr-1Mo steel) [1-2]. The RAFM steels are promising materials for use as the structural component of blanket systems of fusion reactors ~~with high thermal efficiency, because of its excellent high temperature mechanical properties~~. Among the examples of the steels developed are F82H steel, JLF-1 steel, and other steels [3]. In recent years, 9Cr-ODS ferritic steels, which is an advanced option of RAFM by strengthening with oxide particle dispersion, have been developed. They are known to have drastically enhanced creep properties by suppressing dislocation glide, owing to the densely dispersed nano oxide particles in the matrix [4]. According to the past results, the attractive properties of these materials are realized by microstructure of tempered-martensitic phase obtained by an N-T (normalized–tempered) heat treatment. These ~~texture-controlled~~ heat treated materials, however, have poor workability, which is one of the critical issues for fabricating the necessary products of the fusion blankets [5].

Bonding can be a solution to fabricate products out of materials with poor workability. A number of techniques have been utilized for bonding RAFM, such as electron beam welding, friction welding, diffusion bonding [6-7], etc. However, it is possible that the controlled microstructure of the RAFM materials is lost near the bonding interface, inducing degradation of toughness in welded joint[8]. Reheating treatment, which can recover the microstructure of RAFM, is, however, difficult to apply to the all blanket components after fabrication.

As a solution of this problem, we have focused on “Super plasticity.” The Super-plasticity can be classified as “micro-grain super plasticity” based on grain sliding and “transformation super plasticity” based on high temperature dislocation-creep deformation. The SPF is generally known as a formation technology using a macro-deformation by grain sliding (micro-grain super plasticity phenomenon) [9].

As a breakthrough idea, superplastic forming (SPF) based on a transformation super plasticity (TSP) phenomenon was suggested. ~~On the other hand, there are a TSP phenomena based on high temperature dislocation-creep deformation.~~ The TSP deformation takes place by macro-elongation during cyclic temperature variation induced by a phase transformation. The phase transformation generates dislocations around the new phases according to the difference in the specific volume between the two phases [10-13]. In this process, the deformation progresses continuously because dislocations are continuously generated by the cyclic temperature variations which climb according to the stress induced by the phase change.

The TSP phenomenona have not yet been applied for working RAFM steels. The application is considered to be innovative in the working process of RAFM, providing more freedom to the blanket design. The purpose of the present study is to investigate the TSP phenomenona in RAFM steels, and evaluate the effect of cyclic temperature variations on their deformation properties.

2. Experimental Procedures

2.1. Materials for tensile test

In the present study, JLF-1 steel and 9Cr-ODS steel were compared as a typical RAFM steel and a RAFM ODS steel. The composition of the two materials are listed in Table 1. The JLF-1 steel, which was fabricated via casting - hot rolling by NIPPON STEEL & SUMITOMO METAL Co., was subjected to solution heat treatment (1050 ˚C /1 hr) and tempering(780 ˚C /1 hr) [14]. The 9Cr-ODS steel, which was fabricated via mechanical alloying (MA) – hot extrusion – hot forging by KOBELCO Res. Inst. , was subjected to normalizing (1050 ˚C /1 hr) and tempering (800 ˚C /1 hr) [15].

2.2. Heat treatments

The JLF-1 steel and the 9Cr-ODS ferritic steel, containing 0.1 and 0.13 mass % C, undergo the γ→α phase transformation in the temperature range of 850-920 ˚C and 880-920 ˚C, respectively ~~[4,16]~~. These temperature were determined by dilatometric measurement on studies of reference [4, 16]. In the present study, the cyclic temperature variation was carried out between 800 and 1000 ˚C to induce the transformation for both steels, can cover phase transformation temperature completely. Fig.1 shows an illustration of the apparatus for the tensile test with a cyclic variation of temperature. The tensile specimen was heat-treated using the furnace vertically movable to obtain the heat cycling. The heating and the cooling were carried out by the vertical up movement of pre-heated furnace and the vertical down movement, respectively. The heating and cooling rate depended on temperature of the pre-heated furnace and the air temperature, respectively. The heating rate was set 1.6 ˚C/s by using a pre-heated furnace (T>1000 ˚C), and the cooling rate was set 6.0 ˚C/s.

2.3. Tensile tests with constant load

The specimens for tensile tests were machined by wire electro-discharge methods. The specimen dimension for the tests was 4 mm x 16 mm x 0.5, 1.2 mm, as shown Fig.2 (a). The grip section of the specimen was cramped by a specialized jig which can support the tension by the shoulders of the specimen, and the gage of specimen was loaded at 0.87 kg/mm^2 (1.25 kg), 1.74 kg/mm^2 (2.50 kg)[12)] and 3.47 kg/mm^2 (5.00 kg). The specimen temperature was directly measured by a thermocouple, as shown Fig.2 (b).

2.4. Tensile tests with a varying load

In general, simplification of the necessary procedure in the manufacturing processes enhance their efficiency. In the present tensile test condition, it was predicted that temperature cycles for more than 100 times are needed for obtaining TSP if the load is kept constant [12)]. For the purpose of decreasing the number of necessary temperature cycles, tensile tests with varying load from 3.47 to 0.87 kg/mm^2 were also carried out. In this experiment, the first step of the test was carried out with 3.47 kg/mm^2 load for 5 temperature cycles, followed by the second step with 1.74 kg/mm^2 for 60 cycles, and the final step which was continued to the fracture, with 0.87 kg/mm^2 for 67 cycles.

3. Results and Discussions

3.1. Tensile test of JLF-1 with 1.74 kg/mm^2 load

Fig. 3 is the result of the tensile tests with 1.74 kg/mm^2 load, which shows the effect of macro deformation by the cyclic temperature variation between 800 and 1000 ˚C. The specimen fractured after 69 cycles, when the elongation reached 172 %. For comparison, the tensile test in an isothermal condition at 1000 ˚C was carried out for 3.1hr, which is comparable to the time for the 69 cycles temperature variations. The result of the test did not exhibit macro deformation with the elongation of only 20 %. These results confirmed that the macro deformation is attributable to the phase transformation between ferrite phase and gamma phase, which was induced by the cyclic temperature variation.

Fig.4 shows microstructure of JLF-1 for the loads 1.74 kg/mm^2 with and without deformation of the test. The JLF-1 microstructure exhibited no changes of the temper-martensite structure and the grain size after deformation by tensile test.

Fig. 5 shows cross section of the specimen after a tensile test with cyclic temperature variation for 69 cycles between 800 and 1000 ˚C. The specimen fractured with the configuration of chisel point type. This means that the deformation was highly ductile. The high ductility was also evidenced by the formation of dimples on the fracture surface.

3.2. Tensile test of JLF-1 for the loads of 3.47, 1.74, and 0.87 kg/mm^2

Fig.6-(a) shows the elongation as a function of the number of temperature cycles between 800 and 1000 ˚C for JLF-1 for the loads of 3.47, 1.74, and 0.87 kg/mm^2. The tensile specimen which was loaded to 3.47 kg/mm^2 fractured after 7 cycles, which exhibited the elongation of 114 %. At one cycle before the fracture, the change of elongation was 53 %, showing a rapid deformation before the fracture in the case of high stress load. On the other hand, the tensile specimen which was loaded to 0.87 kg/mm^2 fractured after 175 cycles, exhibiting elongation of 170 %. At one cycle before fracture, the change of elongation was 29 %, showing a moderate deformation before the fracture in the case of low stress load.

It should be noted that the test with high stress load (3.47 kg/mm^2) has the advantage of high deformation rate, thus short processing time, but the test with low stress load (0.87 kg/mm^2) has the advantage of controllable deformation before fracture.

3.3. Tensile test of JLF-1 with variation of load from 3.47 to 0.87 kg/mm^2 load

Fig.6-(b) shows the load variation experiment with three loads (0~5cycle: 3.47 kg/mm^2, 5~60cycle: 1.74 kg/mm^2, 60~67cycle: 0.87 kg/mm^2). The curve exhibits that the test with variation of the load can reduce the total number of cycles relative to that for the cyclic test with 0.87kg/mm^2 load only, and that the test with a gradual increase in the deformation from 60 to 67 cycles resulted in the increase in the elongation relative to that for the cyclic test with 1.74kg/mm^2 load only

The reduction of the total number of cycles for TSP process is expected to contribute to reducing the processing time of the SPF based on TSP. The reduction of the processing time was various advantages, such as suppressing the contamination during the process.

The load variation may also increase the total deformation. In conventional SPF process, the rate of deformation is controlled by adjustments of normal stress and shear stress for the purpose of avoiding rapid deformation which can induce fracture. The tensile sample with 1.74 kg/mm^2 load fractured with a rapid deformation of 58 % in the last test. On the other hand, tensile tests after change of the load from 1.74 kg/mm^2 to 0.87 kg/mm^2 fractured with gradual deformation of 13 % per cycle (60~67 cycles). Thus the tensile tests with load variation can inhibit rapid deformation to the fracture, which means better controllability of the process, and reach higher elongation than those without the variation.

3.4. Tensile test of 9Cr-ODS ferritic steel on 1.74~8.33 kg/mm^2 load

Fig.7 shows the deformation due to temperature cycling between 800 and 1000 ˚C as a function of the number of cycles for 9Cr-ODS ferritic steel with different loads from 1.74 to 8.33 kg/mm^2. The tensile specimens which were loaded with 1.74 and 4.17 kg/mm^2

did not fracture up to 210 cycles. Because of the small elongation with 1.74 and 4.17 kg/mm^2, tensile tests with 8.33 kg/mm^2 load were carried out with the same temperature cycles. In this case, the specimens fractured with elongation of 13.6 % after 27 cycles.

Fig.8 shows the cross section after the tensile test with heat treatment of 27 cycles between 800 and 1000 ˚C, and with a constant load of 8.33 kg/mm^2. Dimple was observed at the cross section. The primary difference of 9Cr-ODS ferritic steel from JLF-1 steel is the presence of nano-oxide particles which can inhibit dislocation motion which is necessary for creep deformation. The fact that 9Cr-ODS ferritic steel did not exhibit macro-deformation by tensile tests with cyclic temperature variation suggests that the principal mechanism of TSP is the high temperature creep because 9Cr-ODS ferritic steels are much more resistant to thermal creep in the temperature region of the present experiments.

5. Conclusion

JLF-1 steel and 9Cr-ODS ferritic steel are promising materials for fusion reactors. However, these steel have the common disadvantage of limited workability. In this study, an innovative processing using a TSP phenomenon was proposed for improvement of their workability. In this paper, tensile tests with cyclic temperature variation between 800 ˚C and 1000 ˚C, which was intended to cause cyclic transformation between alpha and gamma phases, was carried out. The main results are as follows:

1. The tensile tests on JLF-1 with constant load of 0.87 to 3.47 kg/mm^2 and under cyclic temperature variation exhibited macro-elongation to 172 %. However, tensile test on JLF-1 with constant load (1.74 kg/mm^2) in isothermal condition did not exhibit macro-

elongation. The result showed that this macro-elongation is caused by deformation induced by phase transformation.

2. Elongation per 1 cycle for 3.47 kg/mm^2 was much higher than that for 0.87 kg/mm^2.

3. Tensile tests of JLF-1 with variation of the load from 0.87 to 3.47 kg/mm^2 load (0~5cycle: 3.47 kg/mm^2, 5~60cycle: 1.74 kg/mm^2, 60~67cycle: 0.87 kg/mm^2) showed that the elongation reached to 199 %, and the total number of cycles decreased relative to that for the cyclic test with 0.87kg/mm^2 load only. This suggests that the total deformation increases and the total number of cycles decreases necessarily for the macro-deformation, by applying the load variation. The load variation can also enhance the controllability of the process.

4. On the other hand, the 9Cr-ODS ferritic steel did not exhibit the macro-elongation in the same test conditions, but fractured with elongation of 13.6 % after 27 cycles. Considering the difference of resistance to creep of the two alloys, it is expected that the principal mechanism of TSP is high temperature creep. It was inferred that the increased resistance of thermal creep by nano-oxide particles in the matrix inhibits super plasticity deformation for 9Cr-ODS.

5. Acknowledgment

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References

1) M.Tamura et al., Development of potential low activation ferritic and austenitic steels, J. Nucl. Mater.141-143(1986)1067-1073

2) Y Seki et al., Impact of low activation materials on fusion reactor design, J. Nucl. Mater.

258-263 (1998) 1791-1797

3) A.Kohyama et al., R&D of low activation ferritic steels for fusion in Japanese universities, J. Nucl. Mater. 212-215 (1994) 684-689

4) S.Ukai et al., Development of 9Cr-ODS Martensitic Steel Claddings for Fuel Pins by means of Ferrite to Austenite Phase Transformation, J. Nucl. Sci. and Tec., 39 (2002) 778–788

5) K.Shiba et al.,JAERI Tech 97-038 (1997).

6) H.Fu et al., Microstructural Evolution of 9Cr-ODS and JLF-1 in a HIP Joint after Post-bond Heat Treatment, J. Plasma Fusion Res. SERIES, 11 (2015)

7) K.Shiba et al., Reduced activation martensitic steels as a structural material for ITER test blanket, J. Nucl. Mater. 329-333(2004)243-247

8) D.Y.Ku et al., TIG and HIP joining of Reduced Activation Ferrite/Martensitic steel for the Korean ITER–TBM, J. Nucl. Mater. 417 (2011) 67–71

9) A.J. Barnes, Superplastic Forming 40 Years and Still Growing, JMEPEG 16(2007) 440–454

10) R. H. Johnson & G.W. Greenwood, Deformation of uranium during alpha /beta cycles under small stresses and a quantitative interpretation of the mechanical weakness of metals undergoing phase transformations, Nature 195 (1962).

11) L.F. Porter, P.C. Rosenthal, Effect of applied tensile stress on phase transformations in steel, Acta Met., 7(1959), 504.

12) D. Oelschlagel, V.Weiss, Superplasticity of steels during the ferrite-austenite transformation, Trans. A.S.M., 59(1966), 143-154.

13) F. W. Clinard & O. D. Sherby, Strength of iron during allotropic transformation, Acta Metal, 12(1964), 911-919.

14) Y.F. Li et al., Effect of thermal ageing on tensile and creep properties of JLF-1 and CLAM steels, Journal of Nuclear Materials 386-388 (2009) 495–498

15) T. Muroga et al., Fabrication and characterization of reference 9Cr and 12Cr-ODS low activation ferritic/martensitic steels, Fusion Engineering and Design, 89 (2014) 1717–1722

16) A.Alamo et al., Physical metallurgy and mechanical behaviour of FeCrWTaV low activation martensitic steels: Effects of chemical composition, J. Nucl. Mater. 258-263 (1998) 1228-1235

Table 1 The materials as received and experimental conditions.

Materials	Supplier	Composition (wt.%)	Transformation Temp.(˚C)	Cyclic Temp.(˚C)	Cyclic rate
RAFM steel (JLF-1 steel)	NIPPON STEEL & SUMITOMO METAL Co.	Fe-9Cr-2W-0.10C-0.5Mn-0.2V-0.08Ta	850/920 (γ/α)	800/1000	Heating rate: 6.0 ˚C/s Cooling rate: 1.6 ˚C/s
9Cr-ODS ferritic steel	KOBELCO Co.	Fe-9Cr-2W-0.13C-0.2Ti-0.35Y_2O_3	880/920 (γ/α)	800/1000	

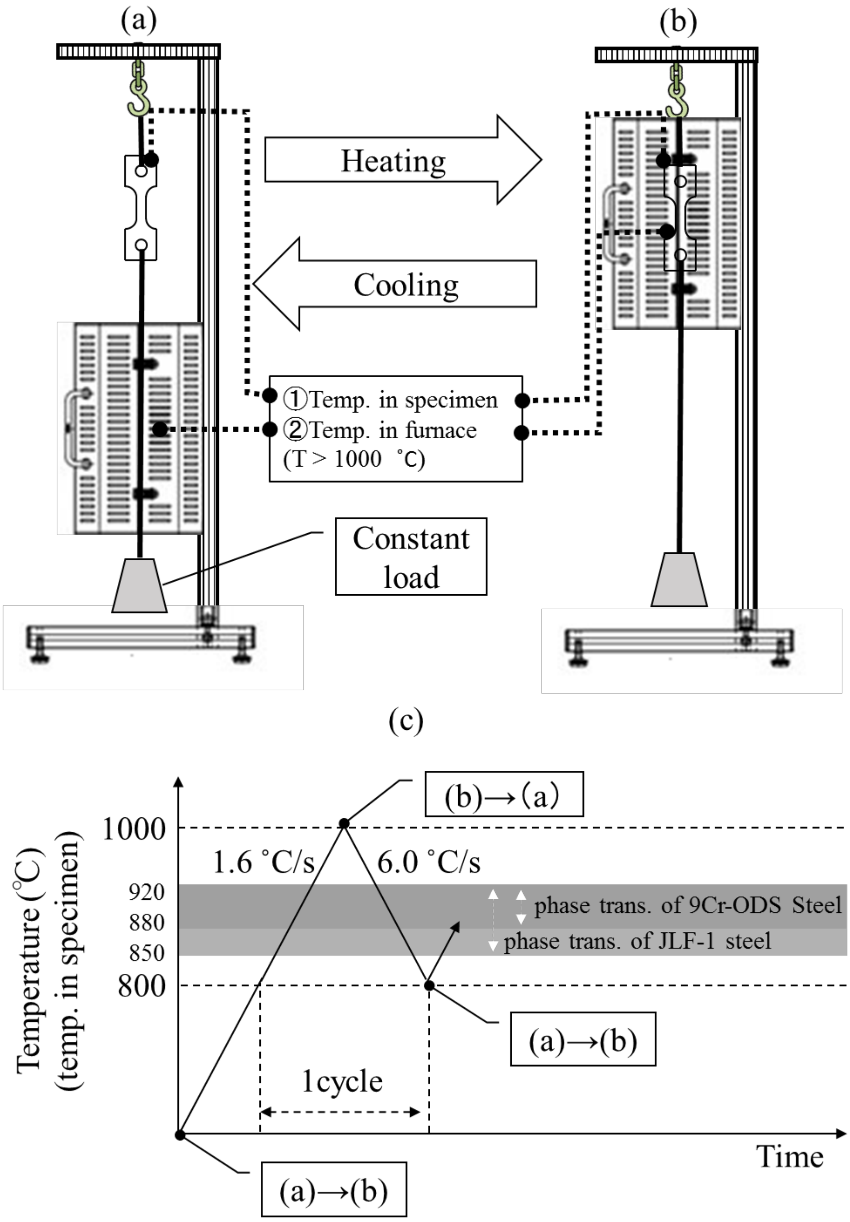

Fig.1 Illustration of the tensile test device.

(a) During cooling.

(b) During heating.

(c) Temperature variation with movement of tensile test device

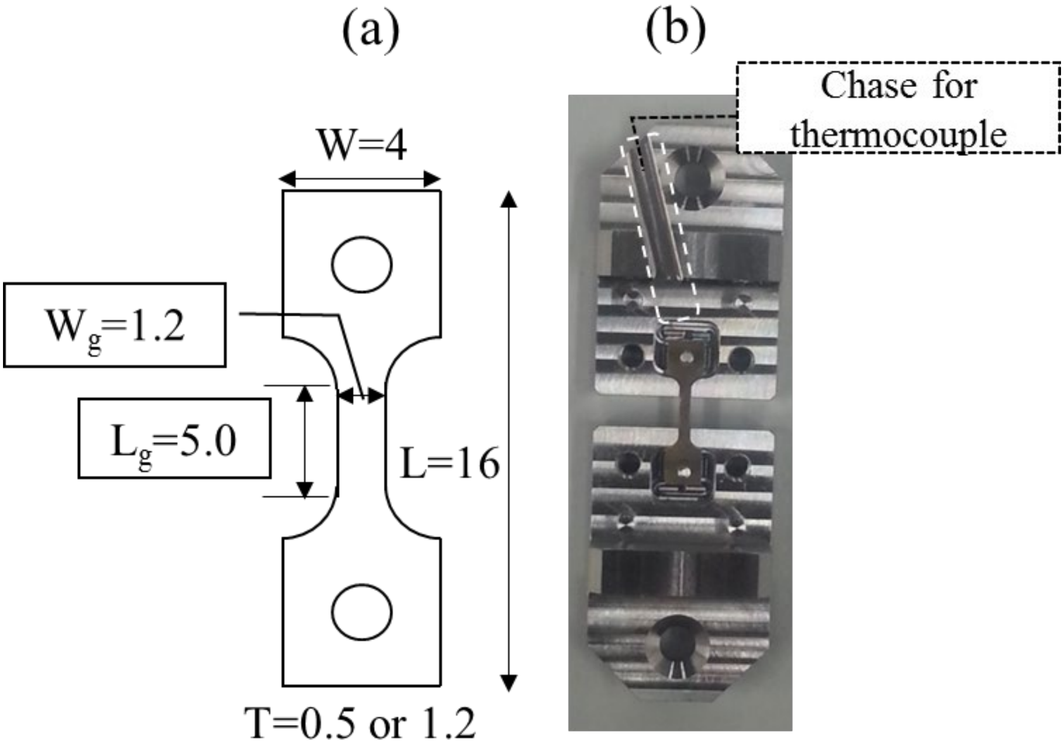

Fig.2 Cramping method of the tensile specimen.

(a) Dimension of the specimen for tensile test.

(b) Jig for cramping.

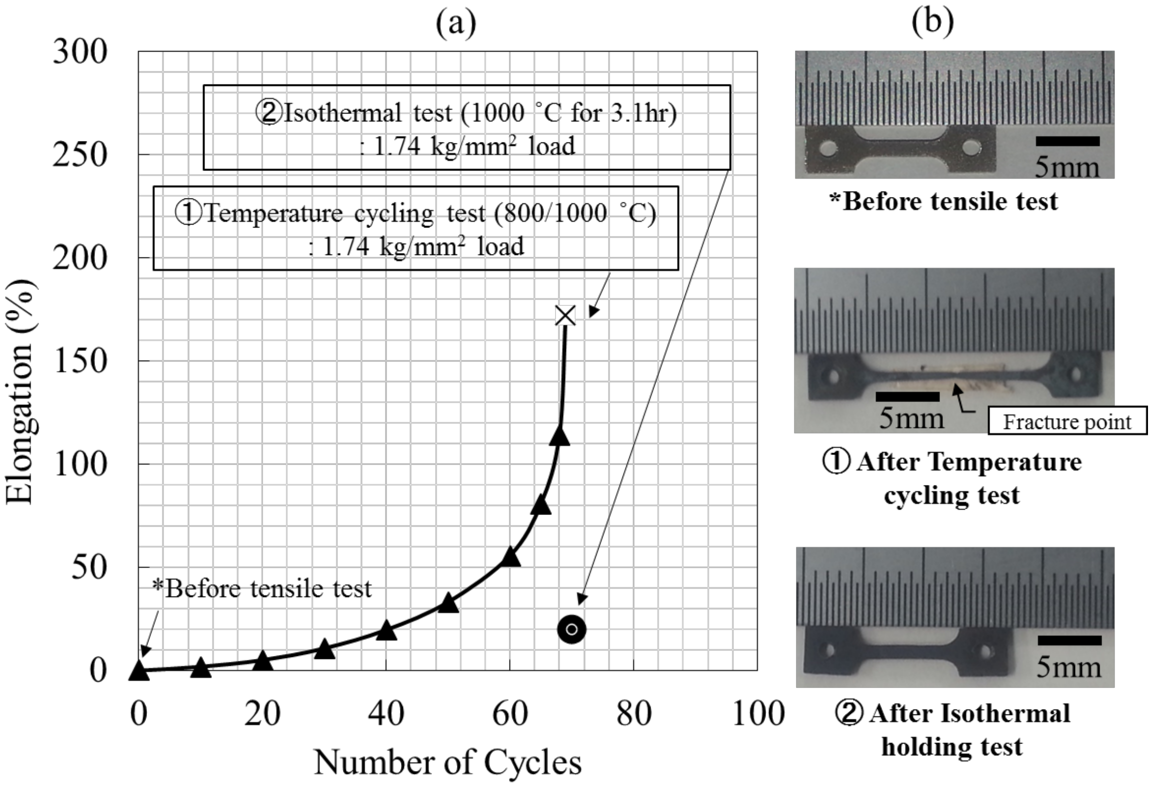

Fig.3 The effect of cyclic heat treatment on macro deformation.

(a) Deformation under temperature cycling between 800 and 1000 ˚C, and isothermal deformation at 1000 ˚C for 3.1hr (constant load of 1.74 kg/mm^2).

(b) The specimens before and after the tests.

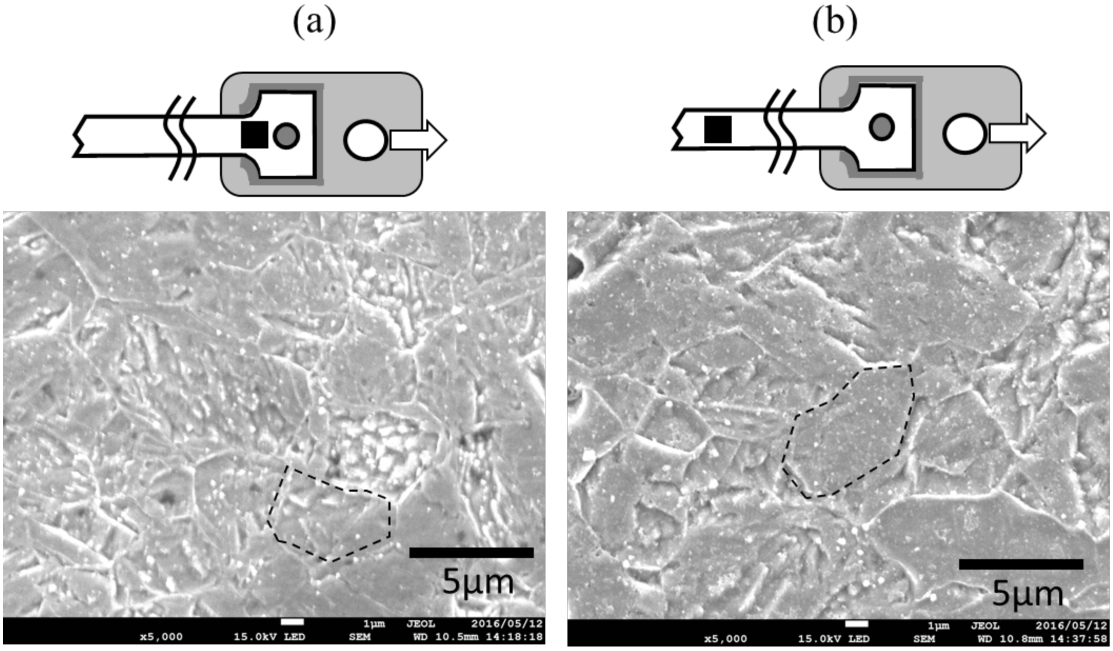

Fig. 4 Microstructure of JLF-1 with and without deformation of temperature cycling test

(800/1000 ˚C): 1.74 kg/mm2 load.

(Broken line is typical grain size)

(a) Without deformation.

(b) With deformation.

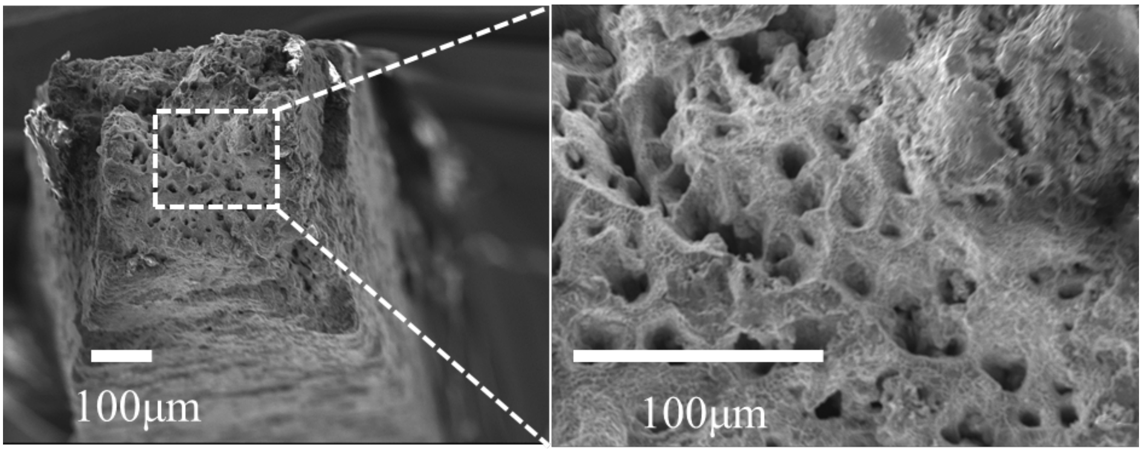

Fig.5 Fracture surface of the sample after the tensile test with temperature variation for 69 cycles between 800 ˚C and 1000 ˚C.

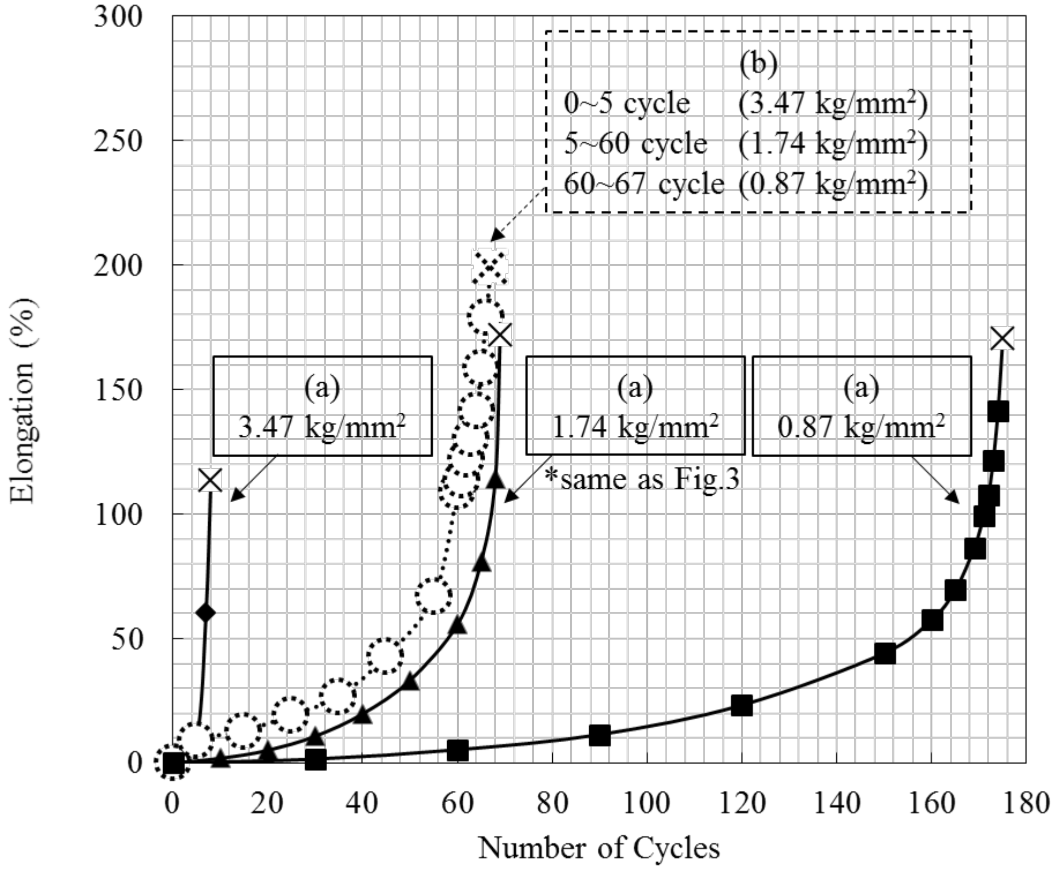

Fig.6 Deformation due to temperature cycling between 800 ˚C and 1000 ˚C as a function of number of cycles for JLF-1.

(a)Elongation behavior to the fracture for three constant loads (0.87, 1.74, and 3.47 kg/mm^2): solid line.

(b)Effect of variation of load from 0.87 to 3.47 kg/mm^2 on deformation. (0~5cycle: 3.47 kg/mm2, 5~60cycle: 1.74 kg/mm2, 60~67cycle: 0.87 kg/mm^2): broken line.

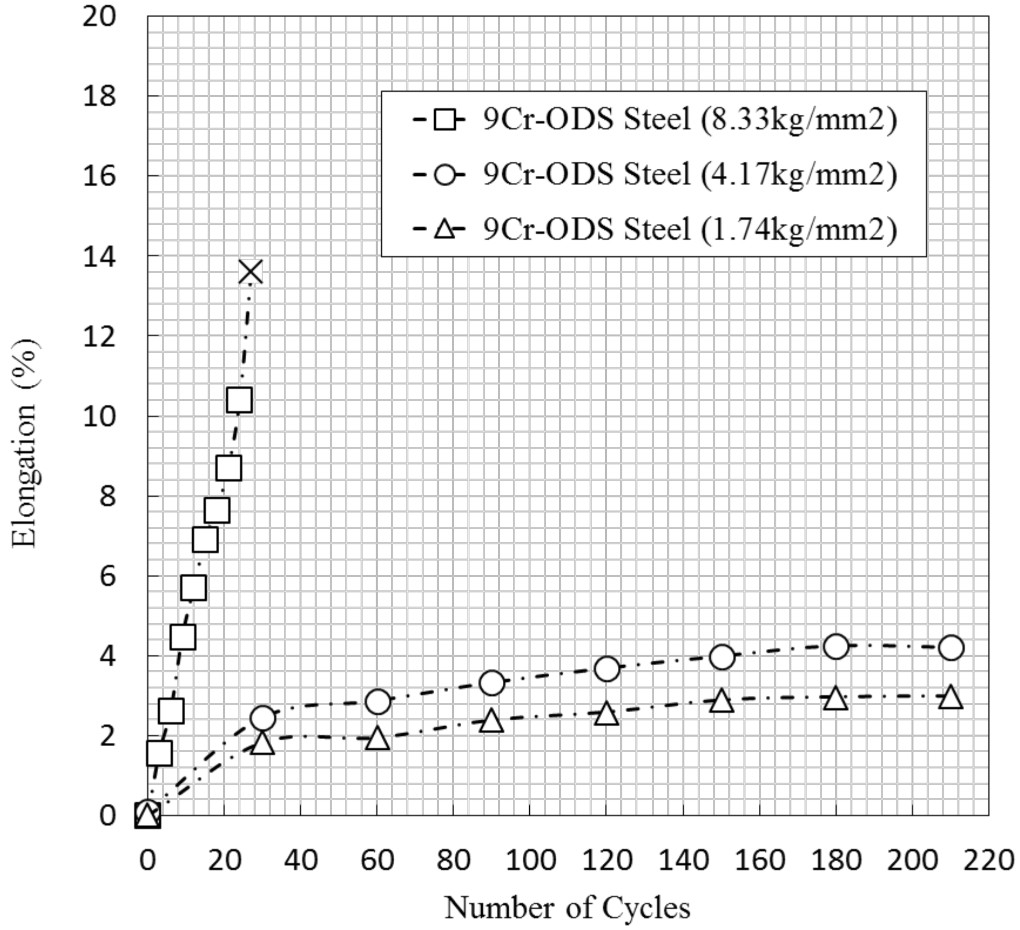

Fig.7 Deformation due to temperature cycling between 800˚C and 1000 ˚C as a function of number of cycles for 9Cr-ODS steel (constant load of 1.74, 4.17 ,and 8.33 kg/mm^2).

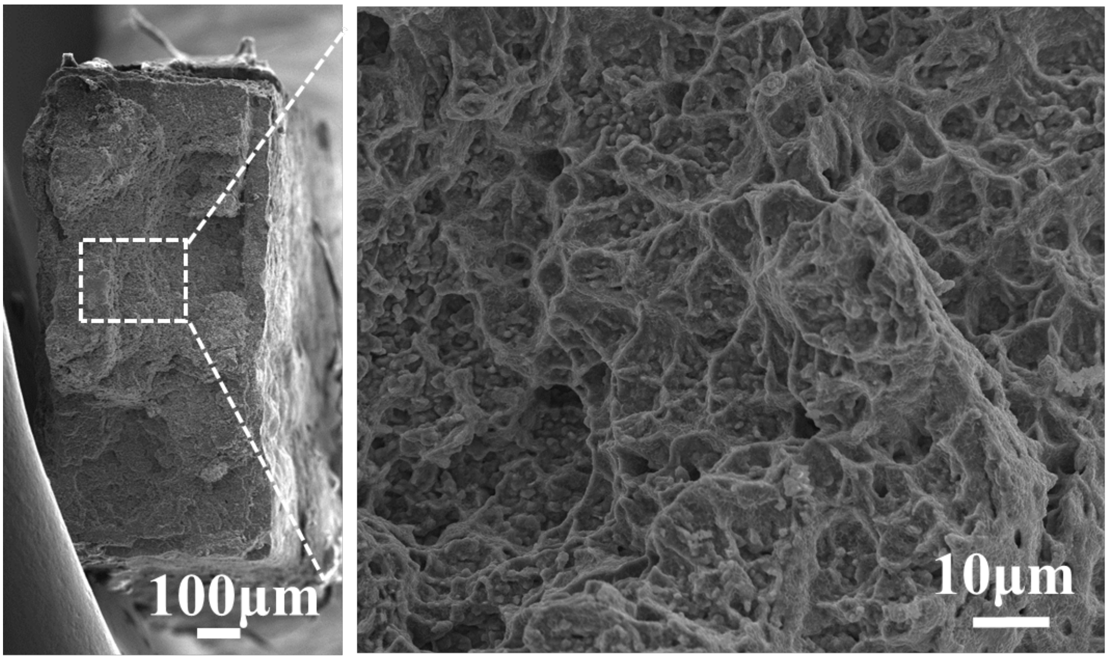

Fig.8. Fracture surface after tensile test with temperature variation of 27 cycles between 800 ˚C and 1000 ˚C. (Constant load of 8.33 kg/mm^2)