Transactions of the Korean Nuclear Society Spring Meeting
Jeju, Korea, May 7-8, 2015

Mechanical Properties of Ni-base ODS Alloy Influenced by Process Variables

Y.B. Chun [a*], B.Y. Lee [a,b], M.S. Kim[a], X. Mao [a], J. Jang[a]
[a]*Nuclear Material Div., Korea Atomic Energy Research Institute, Daejeon, Republic of Korea*
[b]*Department of Nano Materials Engineering, Chungnam National University, Daejeon, Republic of Korea*
[*]*Corresponding author: youngbumchun@kaeri.re.kr*

1. Introduction

Nickel based alloys are considered the reference candidate materials for high temperature gas-cooled reactors which should work under very high harsh conditions, i.e., temperatures above 900 °C and internal gas pressures higher than ~8 MPa [1]. According to a recent investigation, no proven industrial technology could be directly used for such applications. For example, extensive work on Alloy 617 which is the candidate material for the intermediate heat exchanger (IHX) in very high temperature reactors (VHTR) shows that Alloy 617 exhibit quite good creep properties [2-4], the maximum service temperature of Alloy 617 is much less than that required for the VHTR-IHX applications. In this regard, oxide dispersion strengthened (ODS) materials have received a great attention owing to their excellent mechanical properties at higher temperatures, e.g., creep resistance. As part of an alloy development program for nickel base ODS alloy, we have produced an ODS Alloy 617 via mechanical alloying and hot extrusion, and characterized its microstructural evolution during the process and evaluated mechanical properties at elevated temperatures. The current work reports the effects of process variables and yttria contents on the microstructure and mechanical properties of ODS Alloy 617.

2. Methods and Results

2.1 Experimental procedures

An ODS Alloy 617 with the chemical composition given in Table 1 was produced based on a powder metallurgy method and hot extrusion. Prealloyed powder with the chemical composition the same as the conventional Alloy 617 was produced by atomization. A mixture of the prealloyed powder and 0.6 wt.% yttria was mechanically alloyed (MA) by milling with steel balls (steel ball to powder ratio is 15:1 in weight) at 200 rpm for 40 h. in a Simoloyer CM08 horizontal mill. The MA powders thus prepared were sealed in a mild steel container and degassed at 400 °C for 1h under a vacuum of 10^{-4} torr. The MA powders was pre-heated at 1100 °C for 2 h and then hot-extruded with a reduction ratio of 6.25:1. The extruded bar was then solution treated at temperature between 1100 °C and 1350 °C for 2 h. This is reference process route and ODS Alloy 617 fabricated by the route is designated as 'Ref'.

Influences of process variables such as increasing extrusion ratio to 9:1 (designated as 'ER9'), addition of hydrogen reduction in the fabrication process (designated as 'H2'), and the reduction of yttria contents to 0.45 wt.% (designated as '45Y') were investigated and the fabrication routes involved in the present work is schematically shown in Fig. 1.

Microstructures of the samples were characterized using an optical microscopy and scanning electron microscopy. Tensile tests were conducted at RT, 700 °C, 900 °C and 950 °C, using dog-bone type specimens.

Table I: Chemical composition of experimental ODS Alloy 617 in weight percent.

Cr	Co	Mo	Fe	Al
22	12.5	9	1.5	1
Ti	C	B	Y_2O_3	Ni
0.4	0.1	0.003	0.6	Bal.

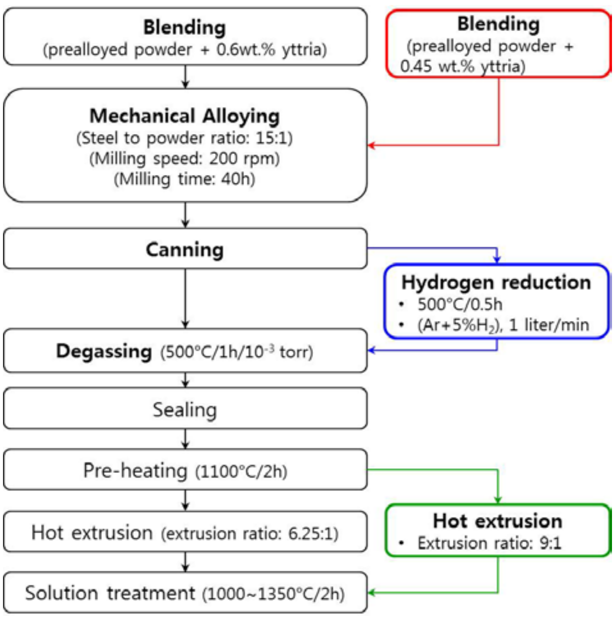

Fig. 1. Fabrication routes for the ODS alloys investigated in the present work. The reference process is shown as black borders. Modified process variables such as yttria contents (red border), hydrogen reduction (blue border), and extrusion ratio (green border) substituted for or added to the reference process.

2.2 Results and Discussion

The microstructures of as hot-extruded ODS Alloy 617 are shown in Fig. 2. There is no significant difference in microstructure between samples with different process variables. All the samples develop very fine grains with an average grain size ~0.5 μm, but are mixed with colonies of coarse grains which are elongated along the extrusion direction. Such coarse grains are thought to develop in the yttria-depleted area, suggesting an inhomogeneity of mechanical alloying. The ER9 sample subjected to a larger amount of extrusion exhibits a slightly finer microstructure.

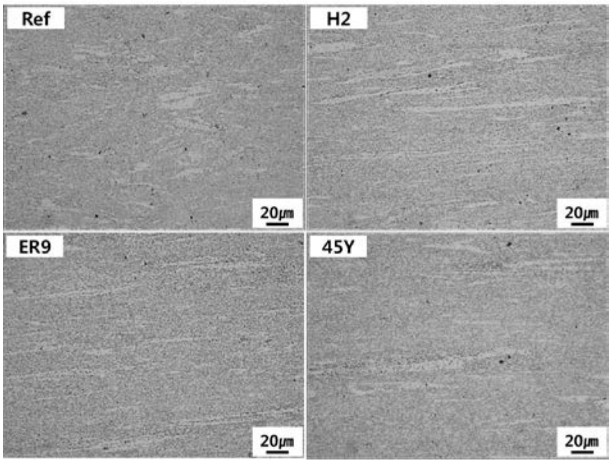

Fig. 2. Optical micrographs of as hot-extruded ODS Alloy 617. The extrusion direction is horizontal.

Solution treatments at up to 1150 °C have little influence on grain structure. However, when solution treated at 1150 °C, the samples start to recrystallize as shown in Fig. 3: new grains form randomly in the microstructure, but such new grains are slightly less in the H2 and 45Y samples, suggesting an effect of oxides or oxygen contents on recrystallization kinetics.

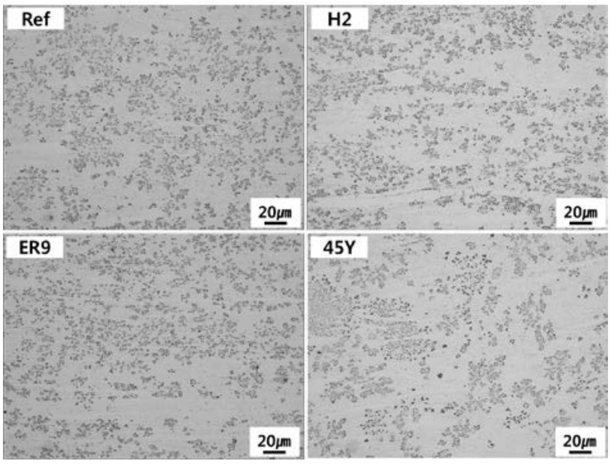

Fig. 3. Optical micrographs of ODS Alloy 617 solution treated at 1200 °C. The extrusion direction is horizontal.

When solution treated at 1300 °C, the deformed matrix is replaced fully with recrystallized grains (Fig. 4), implying a termination of recrystallization. Carbides still remain in the whole microstructure but mainly along the grain boundaries. The 45Y sample shows slightly coarser grain structure.

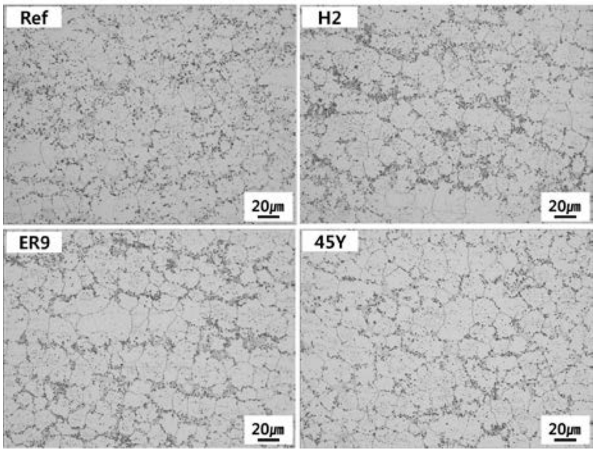

Fig. 4. Optical micrographs of ODS Alloy 617 solution treated at 1300 °C. The extrusion direction is horizontal.

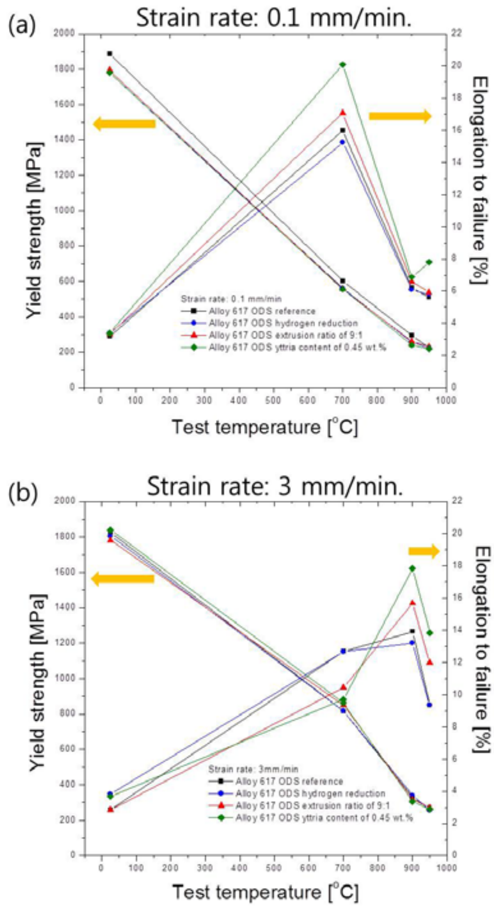

Fig. 5. Tensile properties of as hot-extruded ODS Alloy 617: strained at initial strain rates of (a) 0.1 mm/min and (b) 3 mm/min.

Tensile properties of as hot-extruded ODS 617 strain at initial strain rates of 0.1 mm/min and 3 mm/min are shown in Fig. 5. The yield strengths of the experimental alloys decreased linearly with increasing temperature and the process variables investigated have little influence on the strength. The room temperature yield strength is little affected by the process variables, but higher strain rate results in much higher yields strength at elevated temperatures, esp. at 700 °C. All the samples exhibits negligible ductility (2~3% in total elongation) at room temperature but became ductile at elevated temperatures. It is interesting that samples strained at 0.1 mm/min exhibit the peak ductility at 700 °C, while those at 3 mm/min show peak values at 900 °C. The total elongation at temperature above 700 °C is found to be affected by the process variables: alloy containing less yttria (45Y) and that subjected to a larger amount of extrusion (ER9) shows better ductility than others, which can be attributed to lower oxides contents and less pores, respectively. The results of tensile tests suggest that a slight reduction of yttria content and increasing the amount of hot-extrusion enhance formability at elevated temperature without loss of strength.

3. Conclusions

From the experimental work on the influences of yttria content, and process variables such as hot-extrusion ratio and hydrogen reduction on the mechanical properties of ODS Alloy 617, it is concluded that reduction of yttria contents from 0.6 wt.% to 0.45 wt.% and increasing hot extrusion ratio from 6.25:1 to 9:1 improve the ductility at elevated temperatures without the sacrifice of strength. It is also found that the ductility of ODS Alloy 617 can be controlled by the combination of strain rate and deformation temperature.

REFERENCES

[1] P. Yvon, F. Carré, Structural materials challenges for advanced reactor systems, Journal of Nuclear Materials, Vol. 385, p. 217, 2009.

[2] W-G. Kim, J-Y. Park, I.M. W. Ekaputra, M-H. Kim, Y-W. Kim, Analysis of Creep Behavior of Alloy 617 for Use of VHTR Systems, Procedia Materials Science, Vol. 3, p. 1285, 2014.

[3] G-G. Lee, S. Jung, J-Y. Park, W-G. Kim, S-D. Hong, Y-W. Kim, Microstructural Investigation of Alloy 617 Creep-ruptured at High Temperature in a Helium Environment, Journal of Materials Science and Technology, Vol. 29, p. 1177, 2013.

[4] L.J. Carroll, C. Cabet, M.C. Carroll, R.N. Wright, The development of microstructural damage during high temperature creep-fatigue of a nickel alloy, International Journal of Fatigue, Vol. 47, p. 115, 2013.