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Experimental Study on Thermomechanical Properties of New-Generation ODS Alloys

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***Abstract*—**By using a combination of new technologies together with an unconventional use of different types of materials, specific mechanical properties and structures of the material can be achieved. Some possibilities are enabled by a combination of powder metallurgy in the preparation of a metal matrix with dispersed stable particles achieved by mechanical alloying and hot consolidation. This paper explains the thermomechanical properties of new generation of Oxide Dispersion Strengthened alloys (ODS) within three ranges of temperature with specified deformation profiles. The results show that the mechanical properties of new ODS alloys are significantly affected by the thermomechanical treatment.

***Keywords*—**Hot forming, ODS, alloys, thermomechanical, Fe-Al, Al_2O_3.

I. Introduction

NEW unconventional structure with specific mechanical and physical properties with new application possibilities in some areas of industry can be obtained using conventional materials through innovative technological techniques. A notable improvement in the field was made by J. S. Benjamin at the International Nickel Company (INCO) laboratory: he proposed a new process based on high energy milling powder metallurgy – later called mechanical alloying [1], [2]. This process was introduced to obtain a fine and homogeneous oxide dispersion within a nickel matrix.

Currently, mechanical alloying is still considered to be the most effective process for obtaining fine and homogeneously distributed particles [3], [4]. The volume fraction of dispersed spherical oxides (usually Y_2O_3) is typically below 1% [5], [6], and the oxides typically have a mean size of 5-30 nm [7]-[9]. Based on the thermomechanical analysis, this paper presents the development of new ODS Fe-Al based alloys applicable at high temperatures up to about 1200 °C. The new ODS alloy consists of a ferritic Fe-Al matrix strengthened with 6-10 vol.% of Al_2O_3 particles. The new ODS alloys produced by combination of mechanical alloying [10], [11] and hot consolidation [12], [13] are new types of materials with a promising property spectrum [14], [15]. The experimental program was carried out in order to get a more detailed insight into this new group of materials, to better understand their processing behaviour and their operational properties.

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II. Preparation of ODS Alloys

Mechanically alloyed (MA) powders were prepared in a low energy ball mill developed by the authors, which enables evacuation and filling by oxygen. The MA powders consisting of Fe10wt.%Al matrix and 6-10 vol. % of Al_2O_3 particles were deposited into a steel container of diameter 70 mm, evacuated and sealed by welding. The steel container was heated up to a temperature of 800-900 ºC and rolled by a hot rolling mill to a thickness of 20-25 mm in the first rolling step and then heated up to a temperature of 1100 ºC and rolled to a thickness of 9 mm in the second step. The final production is 6 mm thick sheet of the ODS alloy which is sent to cutting system for preparation of samples. Afterwards, the specimens were cut by water jet to a standard pretested shape.

III. Experimental Procedure

In order to investigate the thermomechanical treatment of specimens, a servohydraulic MTS thermomechanical simulator (Fig. 1) was used, which allows running of various temperature-deformation paths, different numbers of deformation, strain rate and temperatures. It also capable of applying combined tensile and compressive deformation which leads to increase in plastic deformation (and a high dislocation density) in the specimen.

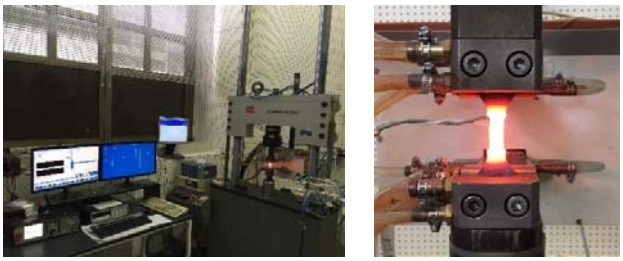


Fig. 1 Treatment on thermomechanical simulator

Four types of material were used in this research as described in Table I. All these materials are based on Fe10wt.% Al ferritic matrix with different particle size and vol.% of Al_2O_3. Al_2O_3 powder was added to prepare the composite which leads to internal oxidation during mechanical alloying and hot consolidation. SEM analysis shows some inhomogeneities which occurred due to sticking of the material during mechanical alloying on the walls of the milling container. It could have some influence on the mechanical properties of materials; however; the mechanical

alloying process is steadily optimized with respect to the homogeneity of the materials.

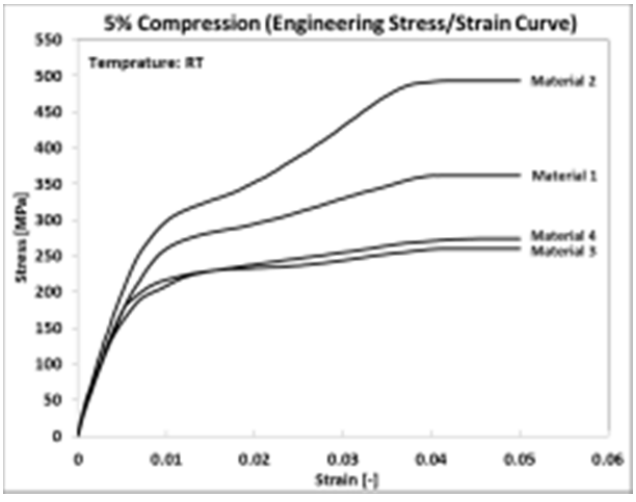
TABLE I
MATERIAL PARAMETERS

Mat. No.	Material Type	Milling time [hours]	Ferritic Matrix	Vol.% of Al_2O_3	Typical Particle Size [nm]
1	Composite	--	Fe10wt.%Al	10	300
2	ODS Alloy	100	Fe10wt.%Al	6	50-200
3	ODS Alloy	150	Fe10wt.%Al	6	50-150
4	ODS Alloy	200	Fe10wt.%Al	6	30-150

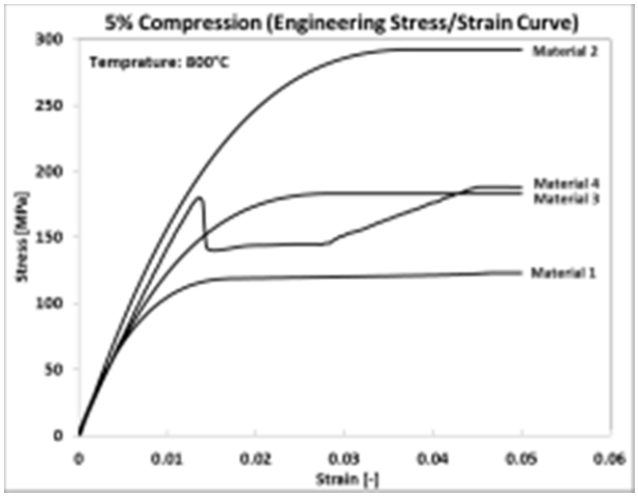
The tests were carried out to investigate the thermomechanical behaviour of the different materials (1 to 4) at different temperatures regarding single loading with constant strain rate of 1 s^{-1}. In order to give a clearer comparison of the results, only the results at room temperature (RT), 800 ºC, and 1200 ºC are presented.

IV. Results and Discussion

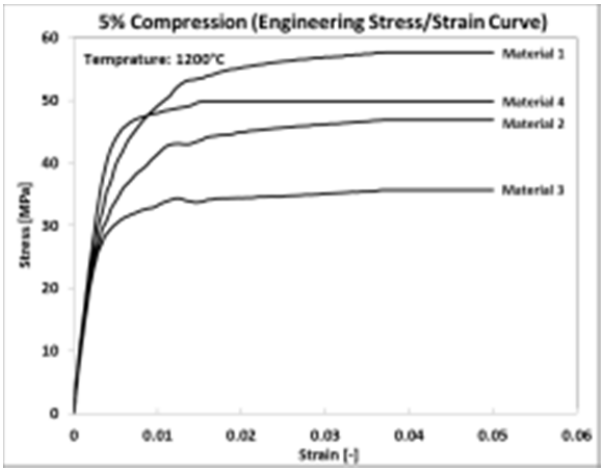
Fig. 2 shows the stress-strain curves for all materials at different temperatures regarding the 5% compression corresponding to treatment number 1. Material 2 exhibits a better strength at 30 °C and 800 °C, but at 1200 °C Material 1 shows a better strength. Hot working behaviour of alloys is generally reflected by flow curves, which are a direct consequence of microstructural changes: the nucleation and growth of new grains, dynamic recrystallization (DRX), generation of dislocations, work hardening (WH), rearrangement of dislocations and their dynamic recovery (DRV). Besides, the flow curve for Material 4 was somehow strange, so it repeated several times; however, it shows the same pattern. It could be concluded that it happens because of the inhomogeneity of the microstructure of this material.



(a)



(b)



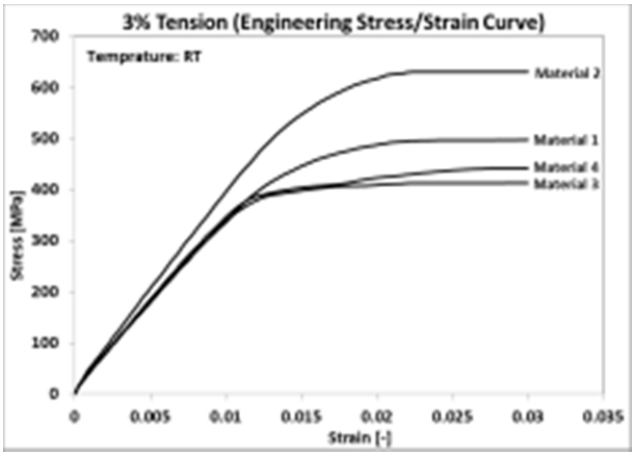
(c)

Fig. 2 Stress-strain curves (5% compression) for (a) RT, (b) 800 °C, (c) 1200 °C

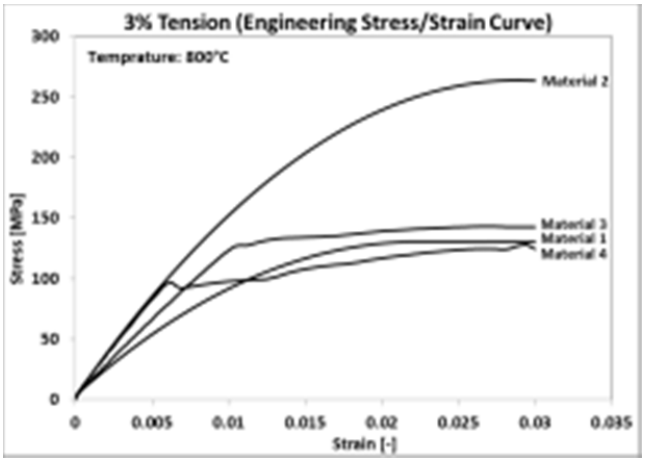
Fig. 3 shows the stress-strain curves for materials 1 to 4 at different temperatures corresponding to the 3% tension. As it can be seen in Fig. 3, material 2 shows a higher strength at 30 °C and 800 °C but at 1200 °C, again material 1 shows a better strength. The maximum stress and trends of curves show sensitivity to the deformation temperatures changes. Thus, by lowering temperature, the yield stress level increased. In the other words, it prevents the occurrence of softening due to DRX and DRV and allows the deformed metals to exhibit WH.

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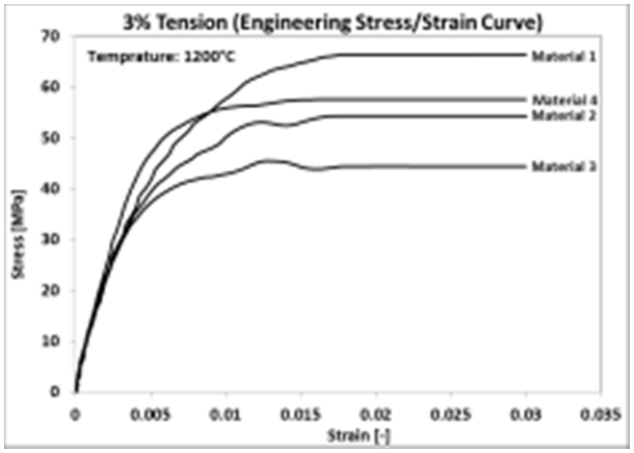
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(a)



(b)



(c)

Fig. 3 Stress-strain curves (3% tension) for (a) RT, (b) 800 °C, (c) 1200 °C

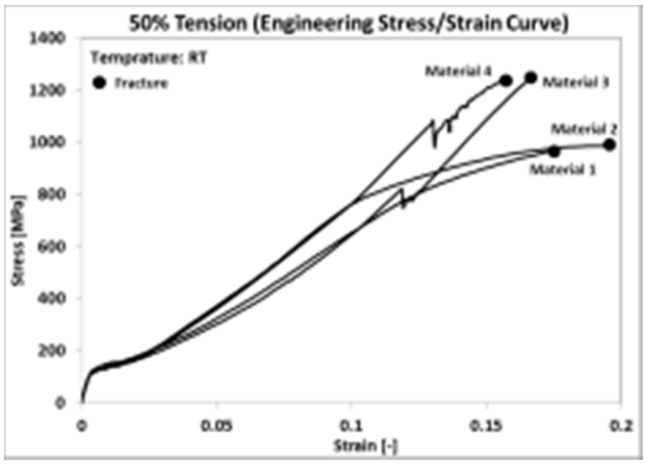
Fig. 4 shows the stress-strain curves for materials 1 to 4 at different temperatures corresponding to 50% tension. Except material 3 and 4, all materials failed below 50% tension at 1200 °C; however, all the four materials failed at RT. It can be seen that Material 2 and Material 4 failed at almost 40% strain at 800 °C the same as Material 1 and 2 at 1200 °C. The trends of flow curves show three distinct stages.

Hardening predominates in the first stage and causes dislocations into stable subgrains. Flow stress exhibits a rapid increase with increasing strain up to a critical value. Then, regarding to large differences in dislocation density, recrystallization happened within subgrains or grains, and new grains are nucleated along the grain boundaries which leads to larger grains.

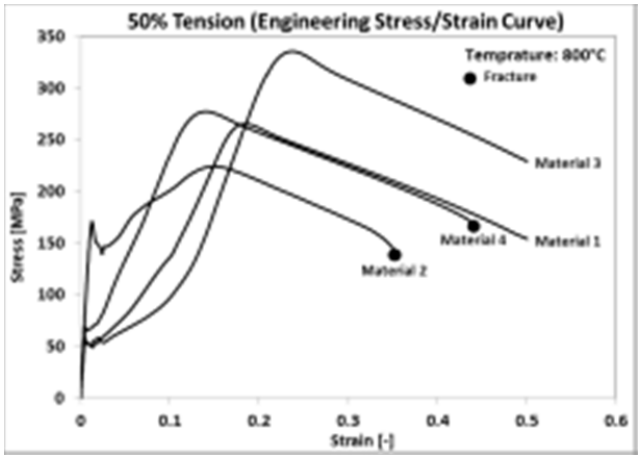
In the next stage, it shows the thermal softening due to DRX and DRV, which becomes more and more important and they nearly compensate hardening.

By the last stage, three types of curves can be recognized:

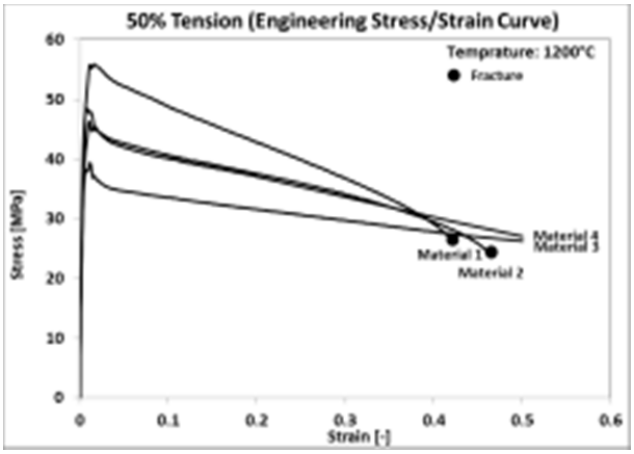
- Increasing continuously with significant work-hardening (Fig. 4 (a))
- Increasing gradually to a steady state with hardening (Fig. 4 (b))
- Decreasing continuously with significant DRX softening (Fig. 4 (c)).



(a)



(b)



(c)

Fig. 4 Stress-strain curves (50% tension) for (a) RT, (b) 800 °C, (c) 1200 °C

V. Conclusion

This paper outlines the results of characterization of the single and multiple deformation thermomechanical behaviour of a new generation of ODS alloys. Four materials differing from each other in the amount and size of the oxides embedded in the ferritic matrix were tested under different conditions. The advantages of all the materials are their low-cost and oxidation-resistance. The oxide dispersion significantly strengthens the material; however, the typical form of the flow curve with DRX softening, including a single peak followed by a steady state flow as a plateau, is more recognizable at high temperatures than at low temperatures. This is because at high temperatures the DRX softening compensates the WH, and both the peak stress and the onset of steady state flow came to lower strain levels. The behaviour of four materials under different heat treatment can be summarized as follows:

1) The maximum stress and trends of curves show sensitivity to the deformation temperatures changes. In the other words, it prevents the occurrence of softening due to DRX and DRV and allows the deformed metals to exhibit WH.
2) All the flow curves show decreasing to a steady state after rapid increase in the stress to maximum value. Differences in softening rate usually depend on DRX and the stress evolution with strain within different stages.
3) In all tests, the elastic strain was higher than plastic strain even for the total stain of 15%, which confirms strengthening the materials within oxide particle effect.

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