Mechanical properties of Fe -10Ni -7Mn martensitic steel subjected to severe plastic deformation via cold rolling and wire drawing

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Abstract. Fe-Ni-Mn martensitic steels are one of the major groups of ultra-high strength steels that have good mechanical properties and ductility in as annealed condition but they suffer from severe inter-granular embitterment after aging. In this paper, the effect of heavy shaped cold rolling and wire drawing on the mechanical properties of Fe-Ni-Mn steel was investigated. This process could provide a large strain deformation in this alloy. The total stain was ε ~7. Aging behavior and tensile properties of Fe-10Ni-7Mn were studied after aging at 753 K. The results showed that the ultimate tensile strength and ductility after cold rolling, wire drawing and aging increased up to 2540 MPa and 7.1 %, respectively, while the conventional steels show a premature fracture stress of 830 MPa with about zero ductility after aging.

1. Introduction
Fe-Ni-Mn Martensitic steels are one of the major groups of ultra high strength steels. They have excellent age hardenability by the formation of nanometer sized inter-metallic precipitates such as f.c.t \( \theta \)-NiMn [1-3]. They suffer from intergranular embrittlement after aging. Discontinuous coarsening of grain boundary precipitates was found to be the main source of embrittlement [4-9]. It is known that grain refinement is an effective way to improve strength and toughness of metallic materials. At present, great attention is paid to the processes of severe plastic deformation (SPD) due to the formation of nano (sub micron grain size) structures upon deformation [9-13]. SPD consists of various methods such as equal channel angular pressing (ECAP) [10-12], accumulative roll banding (ARB)[11-13], high pressure torsion (HPT)[11] and etc. Combination of wire drawing and cold rolling leads to a new method of severe plastic deformation suitable for industrial process, which promotes the work hardening by increasing dislocation density. Most ductile metals can be drawn into ultrafine wire by pulling them through thinner and thinner dies. In most cases, drawing conditions have to be chosen with care: low drawing speeds, low reduction for each pass and diamond dies with low approach angles are recommended [14,15]. Structures obtained during SPD have specific features such as small grains down to submicron and nano level, low density of free dislocations, high angle misorientaiton of these grains and high energy and non-equilibrium state of grain boundaries [11]. These structures lead to changes in the mechanical properties: a significant increase in the strength at good ductility. Effect of ECAP and heavy cold rolling of aged martensitic steels have been studied and

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the resulted microstructure and mechanical properties were discussed [16-17]. It was shown that the aforementioned SPD processes have a major impact on the microstructure and mechanical properties of steels. However, the aim of this paper is to consider the features of nanostructure formation during SPD via shaped cold rolling and wire drawing with focusing on mechanical properties of Fe-10wt % Ni- 7wt % Mn.

2. Experimental procedures

Fe-10wt%Ni- 7wt%Mn was prepared by vacuum induction melting (VIM) and vacuum arc remelting (VAR) routes. The chemical composition of the studied alloy is given in Table.1. The remelted ingot was forged by 50% reduction at 1423 K then homogenized in vacuum furnace at 1473 K for 43200 sec. Heavy shaped cold rolling and wire drawing were carried out to investigate the effect of the large strain plastic deformation on the mechanical properties. Shaped rolling process was carried out in 24 passes. Then the drawing process was carried out with 19 passes with 0.05 mm reduction in diameter at each pass. A final diameter of wire was 0.45 mm. The total applied strain was \( \varepsilon \sim 7 \). Aging behavior was studied at 753 K for different holding time. Microhardness measurements were carried out by Vickers method of 200 g load. Tensile properties were measured using a conventional tensile testing machine with a crosshead speed of 1mm/min. The fracture surface was studied by a scanning electron microscope equipped with EDAX2.

| Table 1 Chemical composition of the alloy (Wt %) |
|---|---|---|---|---|---|---|---|
| Fe | Ni | Mn | C | S | P | N | Al |
| base | 10.50 | 7.00 | 0.006 | 0.007 | 0.005 | 0.005 | 0.003 |

3. Results and discussion

Figure 1 shows a scanning electron microscopic (SEM) micrograph of as annealed sample with fully lath martensite microstructure.

Figure 2 shows the hardness changes in sample A during isothermal aging. The maximum hardness of about 573 HV was obtained after 900 sec. Based on a hardness-aging time curve, the precipitation of f.c.t-\( \theta \) (NiMn) inter-metallic particles is believed to be the main source of hardening [1-3, 19-20]. In two aging time intervals, softening occurred. This phenomenon reported in [21] is attributed to with three main reasons. First, the changes in the dispersion of clusters cause softening. Second, it comes

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from the reverse transformation of martensite to austenite. Thirdly, softening comes from the rearrangement of dislocation substructure leading to a reduction in the total dislocation density. Therefore, for this alloy the hardness decreased to a value of 442 HV after 480 sec and of 362 HV after 2700 sec supposedly due to some of the above phenomena. Figure 3(a) shows mainly brittle intergranular fracture along prior austenite grain boundaries (PAGBs) in the aged-free strain sample where the fracture path is denoted by a dashed white line. The ductile part in fracture surface of the aged wire was cut and shown in Figure 3(b). Small dimples in the fracture surface are perceivable in the magnified picture so the fracture mode is rather defined as the mixed mode of ductile and brittle fracture. The ductile part is responsible for enhanced ductility during the tensile test.

![Figure 3. SEM micrographs (a) brittle fracture in free strain and aged sample (b) ductile part - fracture surface of aged wire](image)

Table 2 shows tensile properties of both samples A and B in as drawn and aged conditions. A total elongation of about 6.9 % and a tensile strength of about 1900(MPa) were obtained for sample A. After aging, there was no important change in mechanical properties. As shown in 1 for sample B, there was a reduction in the UTS with further deformation that could be related to reverse transformation of martensite to austenite [22]. After aging because of more work hardening and more grain refining and formation of finer precipitates, there was a recognizable increase in the UTS and it was the maximum UTS that had been reported for Fe-Ni-Mn alloys yet. A ductility of about 7.1 % was obtained. Heavy cold deformed wire with a total measured strain about $\varepsilon \sim 7$ showed a tensile strength of about 1740 MPa in sample B while as annealed steel shows a tensile strength of about 830 MPa. Sample B revealed a tensile strength of about 2540 MPa after aging with a tensile ductility of $\sim 7.1 \%$.

| Wire diameter (mm) | UTS (MPa) - as drawn | UTS (MPa) - as aged | Ductility (%) |
|-------------------|----------------------|---------------------|--------------|
| Sample A          | 0.60  | 1900            | 2000        | 6.9          |
| Sample B          | 0.45  | 1740            | 2540        | 7.1          |

According to investigations of grain boundary formation during large strain deformation, two different kinds of grain boundaries can be produced: incidental dislocation boundaries (IDBs) and geometrically necessary boundaries (GNBs). These definitions are based on grain boundaries scaling behavior and rate of increase in the average misorientation angle across the boundaries with increasing strain especially in larger strains. Also, the average misorientation angle increases more rapidly with strain for GNBs than that for IDBs. Dislocations in IDBs are redundant whereas in GNBs are nonredunant. Generally presence of GNBs is more effective than IDBs in hardening of microstructure. During wire drawing of bcc materials, grains not only elongate in the drawing direction but also get a
kind of folding. In order to fold the grains over each other, some extra dislocations have to be generated. These dislocations are not annihilated and are defined as geometrically necessary boundaries (GNBs). Formation of GNBs during wire drawing, precipitation density and size can be explained by a significant difference between tensile strengths of samples A and B after aging. It can be estimated that higher GNBs density along with more uniform and fine precipitates in 0.45 mm than 0.60 mm diameter aged wire leads to higher tensile strength in the former than the latter wire.[13-15, 23-24]

4. Conclusions

Combination of cold rolling and drawing is a kind of severe plastic deformation to fabricate nanostructured materials.

Maximum tensile strength was obtained after aging for 900 sec and this tensile strength is the highest value of strength that reported until now in all articles dealt with the examination of Fe-Ni-Mn martensitic steels. The wire exhibits ductile fracture. After aging the fracture surface showed the combination of brittle and ductile mechanisms. It can be concluded that the mechanical properties and ductility were simultaneously improved by severe plastic deformation via cold rolling and wire drawing.

Aging treatment at 753 K for 480 sec and 2700 sec leads to softening.

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