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ScienceDirect
JOURNAL OF IRON AND STEEL RESEARCH, INTERNATIONAL. 2015, 22(4): 324-329

Effects of Different Substrates on Microstructures and Mechanical Properties of a Bulk Nanocrystalline Structure Pure Iron Prepared by Aluminothermic Reaction Casting

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Abstract: A simple method was developed to produce the nanocrystalline pure iron by aluminothermic reaction casting. The microstructure of the iron was investigated by optical microscope (OM), transmission electron microscope (TEM), electron probe micro-analyzer (EPMA), scanning electron microscope (SEM) and X-ray diffraction (XRD). The mechanical performances of nanocrystalline pure iron were tested. It is found that the pure iron consists of nanocrystalline ferrite. For different substrates of copper and glass, the average grain size of the ferrite was 38 and 35 nm, respectively, which is larger on copper substrate than that on glass. The hardness, compressive strength, tensile strength, and total elongation are 167 and 137 HB, 400 and 500 MPa, 243 and 185 MPa, 16% and 10% on copper substrate and glass substrate, respectively. The hardness, tensile strength and total elongation are all larger on copper substrate than those on glass substrate, while the compressive strength is lower. The large supercooling in the product solidification provides the condition for high nucleation rate and thus leads to nano-grained austenite and final nano-grained ferrite transformed from those small austenite grains.

Key words: nanocrystalline; aluminothermic reaction casting; pure iron; substrate; microstructure; mechanical property

The term nanomaterials refers to those materials with grain size between 1 and 100 nm, at least in one dimension[1]. Nanostructured metals and steels can exhibit improved mechanical properties, which is an exciting research theme over the last decades, but the metallic materials in nanometric scale show very low tensile ductility in many studies[2-7]. This is attributed to the existence of a few dislocations in the nanocrystalline grains and a large number of disordered atoms at grain boundaries[8-13]. Such microstructures also show limited room temperature ductility, which can be attributed to the lack of strain-hardening during plastic deformation[14]. Fatigue damage occurred by grain boundary cracking in ultrafine-grained structures, where no slip bands were formed. In the coarse-grained steel, intense slip bands are formed, and crack initiated and propagated along them and grain boundaries. Fatigue resistance can be improved by the grain size refinement[15].

In recent years, researchers have tried to promote the development of iron and steel materials using nano technology. In modern times, iron and its alloy play a very important role in engineering. There has been an ongoing effort in order to obtain nanocrystalline structures in most bulk materials utilizing the following approaches: (1) from large to small, the typical method is severe plastic deformation (SPD) processes, including high pressure torsion (HPT), equal channel angular pressing (ECAP), advanced thermo-mechanical processing, etc.; (2) from small to large, the typical methods include alloying strategies and the spark plasma sintering (SPS), etc. Chen et al.[16] reported that an UNS S32304 duplex stainless steel was obtained by ECPA and subsequent annealing at 973 K, and its uniform elongation increases to 7% with the yield strength of 1100 MPa. Shakhova et al.[17] investigated an S304H steel during bar rolling to a strain of 4 and subsequent annea-

Foundation Item: Item Sponsored by National Natural Science Foundation of China (51164022)
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ling, and this material exhibited a yield strength above 2 000 MPa[17]. In a study performed by Descartes et al.[18], for the pure iron samples processed by high pressure torsion, the yield stress level was increased from initially approximately 200 to 600 MPa, and the grain sizes were refined from initially 100 μm to finally approximately 220 nm.

Although SPD processing can be used to successfully refine the coarse grains of pure metals and alloys to a grain size of a few tens to hundreds of nanometers, most of the desired ductility cannot be reached and for some methods, the experimental equipment is complex owing to the requirement in large strain[19-21]. Hence, traditional techniques are laborious and redundant, and the equipment is complex. Additionally, the method of small to large can lead to impurities easily. For this, a novel conception named aluminothermic reaction was proposed in this study, which can prepare bulk nanocrystalline metal and steel. An advantage of the present approach is simple, low cost and energy consumption, and it is expected to realize industrial application[22-25]. In this study, pure iron was prepared by aluminothermic reaction method and the effect of different substrates on mechanical properties and microstructure was investigated.

1 Experimental Procedure

1.1 Materials preparation

The experimental materials employed were Fe_2O_3 (99% purity), Al (99% purity) and Fe (99.5% purity). 40.3 g Fe powder, 45.3 g Al powder, and 134.4 g Fe_2O_3 powder were weighed according to the stoichiometry of the aluminothermic reaction (1). The reaction powders were dry mixed at a speed of 150 r/min for 8 h using a planetary ball mill with Al_2O_3 spheres. Then, the mixed powders were pressed under a uniaxial pressure of 40 MPa in a copper tube. The thickness of copper and glass substrate is 13 and 10 mm, respectively. Finally, the produced pure iron was added in the reactant powders, respectively. The reactor was purged with argon gas at room temperature and heated to 200 ℃. Then, 5 MPa argon gas was introduced in the reactor and it was heated again to 260 ℃. The lamellar igniter was ignited at 260 ℃ and released exothermic heat resulted in occurrence of the following aluminothermic reaction. The detailed process was reported in literature[22-24].

$$2Al+Fe_2O_3=2Fe+Al_2O_3 \quad (1)$$

1.2 Mechanical properties test

The specimens for mechanical tests were cut by electric discharge machining (EDM). The hardness was tested on an HBRVU-187.5 tester under 294 N. The samples for tensile testing had a rectangular cross-section of 4 mm×2 mm and a gauge length of 16 mm, and specimens were cylindrical of ϕ5 mm×5 mm for compression. All tensile and compressive tests were carried out using a Shimadzu AT10 t test machine at a crosshead speed of 0.2 mm/s at room temperature in air. Tensile testing was conducted at least three times for each specimen from different substrates.

1.3 Microstructure characterization

The phase constitutions of pure iron were identified by X-ray diffraction (XRD) analysis with Cu radiation. The microstructures were investigated under an Olympus Mef3 optical microscope (OM), an EPMA-1600 electron probe microscope, a JEM-6700F field emission scanning electron microscope (SEM), and a JEOL 2010 transmission electron microscope (TEM). The chemical etchant used for OM and SEM observation was a solution of 4% nitric acid alcohol. Thin foils of about 0.8 mm in thickness for TEM observation were first cut by EDM, and then ground to the thickness of approximately 200 μm. Then, the disk of 3 mm in diameter was dimpled and finally polished by twin-jet polishing with a solution of 98% ethyl alcohol and 2% perchloric acid ($HClO_4$) in volume.

2 Results

2.1 Mechanical property

The hardness of the current samples obtained on different substrates in comparison with conventional pure iron is displayed in details in Fig. 1. It is

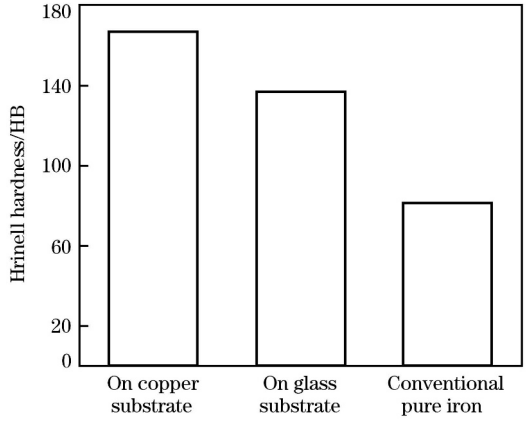

Fig. 1 Hardness of pure iron produced by aluminothermic reaction casting