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Refinement effectiveness of self-prepared (NbTi)C nanoparticles on as-cast 1045 steel

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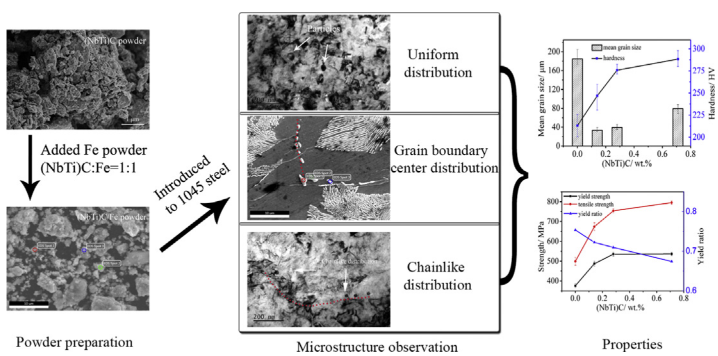
HIGHLIGHTS

- (NbTi)C/Fe complex particles were prepared by the self-designed method.
- Microstructure of the reinforced steel was refined.
- Strength and hardness of reinforced as-cast steels were increased.
- The refinement mechanism of (NbTi)C nanoparticles in 1045 steel has been discussed.

GRAPHICAL ABSTRACT



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ABSTRACT

(NbTi)C nanoparticles were prepared using mechanical alloying (MA) and heat treatment process, and the nanoparticles pre-dispersed with Fe powder were introduced to 1045 steel. The microstructure of powders during different ball milling steps and as-casted steels were investigated using optical microscope (OM), scanning electron microscope (SEM), X-ray diffractometer (XRD) and transmission electron microscope (TEM). The results showed that the added carbides in as-cast steels were mainly composed of three kinds of types: uniform distribution; grain boundary center distribution and chainlike distribution. The uniformly distributed (NbTi)C nanoparticles in the matrix acted as effective heterogeneous nuclei that strongly refined the crystallization microstructure; the grain boundary center distributed particles interacted with the solid-liquid interface, thereby, hindered and restrained the movement of the solid-liquid interface; and the chainlike distributed nanoparticles move with the migration of solid-liquid interface and did not inhibit the interdendritic growth significantly. Compared with the cast steel without (NbTi)C nanoparticles, the modified steels have significantly refined microstructure, improved strength and increased hardness. With the further increase the amount of (NbTi)C nanoparticles, the efficacy of the (NbTi)C nanoparticles for refining crystallization microstructure weakened.

1. Introduction

Microstructure refinement is one of the most effective methods to improve the mechanical properties of cast iron and steel [1]. Nano- and/or micro-sized second phase particles are added to iron and steel melts to modify as-cast microstructure [2–6]. The Second phase

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particles should have high melting temperature and a suitable wettability to ensure a stable and uniform distribution in iron and steel melts; a good interface structure is also required to produce heterogeneous nuclei [7,8]. Nitrides, carbides, oxides or borides with high melting temperature are usually used as modifiers. Ti-carbonitride, V-carbonitride, Nb-carbonitride are the most common second phase particles for refining the microstructure of cast iron and steel because of their unique physical and chemical properties [9–11]. In addition, carbonitrides are common phases in iron and steel. Fine carbonitride particles in as-cast microstructure also exhibit high precipitation strengthening potency, thereby increasing the strength of steels [12,13].

Significant microstructural refinement and crystal morphology alteration are observed because active crystallization nuclei could be created by the introduction of Ti-carbonitride, V-carbonitride, Nb-carbonitride particles in molten Fe-matrix materials [14–16]. However, according to the theory of planar disregistry, VN, VC, TiN, TiC, and NbC can become active heterogeneous crystallization nuclei for δ-Fe phase, and they are not effective heterogeneous crystallization nuclei for γ-Fe phase [17]. Nonetheless, G.F. Liang [18] calculated the heterogeneous nuclei catalysis of certain carbides during non-equilibrium solidification of austenite steel using the improved Thomas-Fermi-Dirac model based on Tillen's electrostatic effect theory. Results showed that VC, NbC, TiC, TaC, ZrC, and CaS have certain heterogeneous nuclei catalytic activity that follows the order TiC > ZrC > CaS > TaC > NbC > VC. Thus, the electrostatic effect of carbide surface for heterogeneous nuclei catalysis is more effective than the planar disregistry. Nano-sized carbide particles may have more potency of becoming the heterogeneous nuclei than micro-sized carbide particles, thus refining the microstructure. High surface energy of the nano-sized carbide particles generates stronger interaction between particles and molten metal, thereby promoting the heterogeneous nucleation [19,20].

Although nano- and micro-sized carbide particles are made on cast iron [21] and some steels with specific application [22], the influence of nano-sized particles on microstructure and properties of cast steels for general application, particularly the exact mechanism for the microstructure refinement and the mechanical properties improvement of the reinforced steels, need further investigate. The nano-sized particles tend to form aggregates and are difficult to distribute uniformly in the molten metal. Therefore, the method of preparing and modifying the surface of nano-sized carbide particles is necessary to improve the homogeneity of the particles distributed in the molten metal.

Based on our previous work [16,23], the present study proposed a new method for improving the microstructure, stability, and density of nano-sized NbC by adding Ti, and surface-modified (NbTi)C nanoparticles were prepared. Furthermore, the influences of (NbTi)C nanoparticles on microstructure, strength, and hardness of the cast 1045 steel were investigated. The experimental data were used to analyze and discuss the refinement mechanism, strength and hardness improvement of the modified cast steel.

2. Experimental procedures

2.1. (NbTi) C nanoparticle preparations

Mechanical alloying (MA) was applied to prepare (NbTi)C nanoparticles and the surface of which was modified with Fe powder using a planetary high-energy ball milling machine. Commercially available Nb powder (purity of 99.5 wt% and particle size ≤ 40 μm), Ti powder (purity 99.5 wt% and particle size ≤ 40 μm), and graphite powder (purity of 99.9 wt% and particle size ≤ 4 μm) were used as raw materials. The stoichiometric ratio of Nb: Ti = 0.91:0.09 was selected to ensure that the density of the prepared powder could approach to that of molten steel. Mixed Nb and Ti powders were sealed in a stainless-steel jar with stainless-steel balls. A ball-to-powder weight ratio of 30:1 and a rotating speed of 280 rpm were adopted. The stainless-steel jar was filled with pure argon gas (99.99 wt%) to prevent oxidation during the MA process. First, the mixed Nb and Ti powders were ball milled for 2 h. Second, graphite at a molar ratio of (Nb + Ti):C = 1:1 was added in ball-milling jar, and the mixed powders were continuously ball milled for 6 h. At the same time, Fe powder (purity 99.5 wt% and particle size ≤ 140 μm) was ball milled totally 8 h in another ball-milling jar, and alcohol was used as the process control agent (PCA) with 1 wt% addition. Third, the same amount of the Fe powder and (NbTiC) were mixed and ball milled for 3 h. Finally, the mixed milled powder was heated to 750 °C for 30 min in a vacuum tubular furnace to obtain the (NbTi)C/Fe composite powder.

2.2. Smelting test

The Fe-matrix materials modified with nanoparticles were prepared in a vacuum inducting melting and casting furnace. The 1045 steel was selected as base metal which was melted in an MgO crucible at a vacuum condition, and then the compacted blocks of nano-sized (NbTi)C/Fe composite powder were added in molten metal. The melt was then smelted and mechanically agitated at 1580–1620 °C for 2 min to mix the powder thoroughly. After smelting, the cast ingot of ∅ 50 mm × 40 mm was poured and air cooling to room temperature. Four ingots with 0, 0.14, 0.28 and 0.71 wt% (NbTi)C nanoparticles were prepared. The samples for microstructure analysis and performance test were taken from the areas pointed by a black arrow in Fig. 1 where the microstructure only consists of equiaxed grains. The area was selected from same location to ensure that the microstructure was only controlled by the added particles. The final chemical compositions of the test steels are listed in Table 1 which was measured by a SPECTRO MAXx (LMX07) direct-reading spectrometer.

2.3. Microstructure characterization, tensile test and hardness test

A D/MAX-2500/PC X-ray diffractometer (XRD) with Cu-Kα radiation from 10° to 120° was employed to analyze the structural changes of mixed Nb, Ti, C, and Fe during MA and heat treatment. The operating voltage was 40 kV, electric current was 200 mA, and scanning speed was 2°/min. The microstructures of the as-cast steel specimens were characterized using a Zeiss Axiovert 200 MAT optical microscope (OM), a S4800 type scanning electron microscope (SEM) equipped with an Energy Dispersive X-ray Detector (EDX) spectrometer (the acceleration voltage is 20 kV and the resolution of probe is 129 eV), and a FEI Tecnai G2 F30 transmission electron microscope (TEM), including

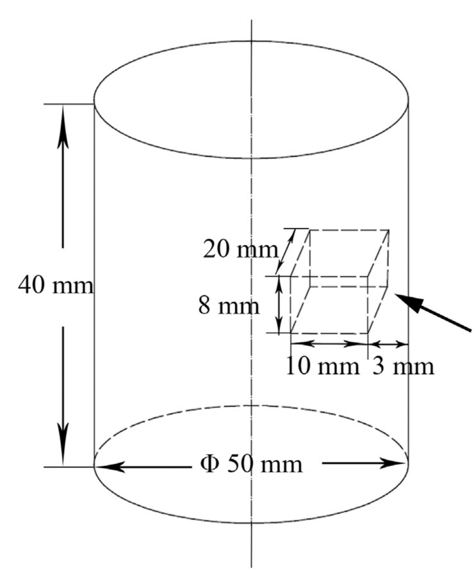

Fig. 1. Schematic of cast with places the samples for metallographic characterization are taken.