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Plastic deformation behavior of functionally graded 9Cr18 steel after thixoforging and heat treatment

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ABSTRACT

The strain hardening behavior and evolution of deformation structure for functionally graded 9Cr18 steel were investigated in the article. Meta-austenite was retained after semi-solid forming, while the liquid was extruded outside to form fine dendrites. Thus the material could demonstrate functionally graded property. Solid austenite grains inside the solid/liquid boundary were connected with each other and would support the high strength with compressive strength of 4680 MPa at compression ratio of 53.2%. The material exhibited obvious four-stage strain hardening characteristics with a long continuous strain hardening stage. The stacking fault energy (SFE) for inner austenite was calculated to be ~24.2 mJ/m^2 and the value fell into intermediate range. The deformation behavior was dominated by planar dislocation, suppression of cross slip and formation of secondary deformation structures (mechanical twinning and ϵ-martensite). The formation of multiple variants of mechanical twinning or ϵ-martensite played a dynamic Hall-Petch effect and the tendency of synchronous improvement of strength and plasticity (SISP) was obtained. The functionally graded property avoided crack formation from brittle eutectic and the material demonstrated a unique ductile fracture characterization.

1. Introduction

Semi-solid forming (SSF) is considered as a conceivable alternative to conventional forming technologies such as casting and forging [1,2]. SSF is an effective near-net-shape forming process to produce components with complex shape, which requires fewer forming steps and lower forming load [3]. In semi-solid temperature range, the microstructure that spheroidal solid particles are suspended in the liquid matrix demonstrates good thixotropic behavior, in which viscosity decreases with the shearing process [4]. As for SSF of steel, there exists complicated phase transformation during SSF and subsequent heat treatment. Researchers attempt to utilize these features to design materials with special functions.

Typically, phase transformation during SSF of steel is determined by chemical composition, processing parameters and heat treatment procedures. For low alloying steel such as 100Cr6, solid austenite particles in semi-solid temperature may transform into martensite structure after fast cooling, while for high alloying steel such as X210CrW12, austenite is stabilized after fast cooling due to its high content of C and Cr alloying elements [5]. The meta-austenite exhibits face centered cubic (*fcc*) structure, which will help improve the mechanical properties. Turteltaub et al. [6] pointed that the presence of austenite in a ferrite-based matrix enhanced the ductility and strength of the steel. Rogal et al. [7] studied deformation behavior of X210CrW12 steel after semi-solid processing, observing strain induced phase transformation and mechanical twinning in the solid austenite. Secondary deformation mechanisms such as $\alpha_{bcc}/\epsilon_{hcp}$-martensite formation and mechanical twinning are typical strengthening behavior for *fcc* based alloys [8–11]. But for semi-solid alloys, co-existing of solid particles and solidified liquid eutectic may bring several problems. The hard and brittle eutectic structure is likely to become the source of crack and weakens the mechanical properties. Rogal et al. [7,12] discovered that crack propagated along the eutectic structure when conducting compression tests for semi-solid X210CrW12 steel. Chen et al. [13] observed that peritectic precipitation along grain boundaries led to cleavage fracture of Ti14 alloy after semi-solid forging. Various heat treatment procedures have been proposed to eliminate the inhomogeneous microstructure [14–16]. In these procedures, the eutectic structure is treated as a kind of defect and the goal is to improve the mechanical properties by providing a more uniform distribution of alloying elements.

The liquid phase may demonstrate special properties and can be used in several situations. Kang et al. [17] pointed that macro separation of solid/liquid phase occurred during thixoforming process. Previous study has shown functionally graded material

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with changes of microstructure and properties can be fabricated through reasonable design [18]. The surface of material will provide high wear resistance and the inner area will support high strength. But the deformation mechanism of semi-solid alloy needs to be clarified. The present study investigates deformation behavior of 9Cr18 thixoforging specimen via compression tests. The strain hardening behavior and evolution of deformation structure are discussed in relation to the whole compression process. The research is aimed at clarifying the relationship among processing, microstructure and property of semi-solid 9Cr18 steel.

2. Experimental procedures

2.1. Functionally graded material

The material used for present study is commercially produced 9Cr18 stainless steel (AISI: 440C martensitic stainless steel) with the chemical composition given in Table 1. Semi-solid billet was prepared by wavelike sloping plate method and then thixoforged through a designed set-up [18,19]. The thixoforging temperature was 1340 °C and the strain rate was 0.02 s^{-1}. After thixoforging, the specimen was tempered at 550 °C for 2 h. A multidiameter shaft shown in Fig. 1(a) was manufactured after thixoforging and heat treatment (T-T-550 specimen). Functionally graded material (FGM) with hard surface and tough center could be obtained due to macro separation of solid/liquid phase. As shown Fig. 1(b), there exists a distinct solid/liquid boundary near the surface of the specimen. Liquid is extruded outside from the center and solidifies to form fine dendrites, while solid austenite particles are retained inside the boundary (Fig. 1(c) and (d)). Solid particles are connected with each other, which is different from conventional semi-solid microstructure that solid particles are suspended in the liquid matrix [7].

2.2. Microstructure observations and mechanical properties analysis

The compression tests were performed using CMT5105 electronic universal experiment machine at a compression rate of 1 mm/min. As shown in Fig. 2, the samples were cut in the inner area of the component, where the solid particles remained. The size of the samples was Φ 4 × 6 mm following the standard of GB/T 7314-2005. Some of the compression tests were performed up to failure of the specimens, while some of the tests were interrupted at predetermined strains (0.05, 0.1, 0.25, 0.50) to characterize the changes of microhardness and the evolution of deformation microstructures. X-ray diffraction (XRD: DMAX-RB, Cu target, operated at 40 kV and 150 mA) was performed to understand the microstructural evolution. Vickers hardness tests were carried out on a HXD1000T tester and the tests were performed in accordance with the standard of ASTM E384-2010e2. Specimens were mechanically polished and etched with Vilella's reagent (1 g picric acid, 100 ml alcohol, 15 ml hydrochloric acid). Then the microstructure was observed using ZEISS Imager.M2m optical microscopy (OM) and ZEISS EVO 18 scanning electron microscope (SEM). JEM-2100 transmission electron microscopy (TEM) was used to observe the microstructure. TEM specimens were grinded to a thickness of 50 μm and electropolished with a twin jet electropolisher at −30 °C (MTP-1A, solution containing 95 vol% C_2H_5OH and 5 vol% $HClO_4$). All the studies of microstructure observation, XRD analysis and hardness tests were conducted on the surface parallel to the compression direction.

Table 1
Composition of the studied 9Cr18 stainless steel (wt%).

C	Cr	Si	P	S	Al	Ni	Co	Fe
0.97	17.33	0.52	0.02	0.005	0.10	0.16	0.12	Bal

3. Results

3.1. Characterization of the multidiameter shaft

Typically, 9Cr18 steel is a kind of martensite stainless steel. The conventional heat treatment (CHT) procedure for 9Cr18 steel consists of quenching at 1050 °C and tempering at 200 °C for 2 h. Hardness of 9Cr18 steel after CHT is about 670 HV (59.2HRC). The phase structure mainly consists of martensite and a small quantity of Cr_7C_3 carbide shown in Fig. 3. The specimen that undergoes thixoforging and heat treatment (T-T-550) demonstrates unique microstructure and properties. As shown in Fig. 1, T-T-550 specimen exhibits FGM property. Liquid is extruded to the surface of specimen and forms fine dendrite structure, while the solid particles are retained inside and connected with each other. According to XRD results in Fig. 3, the solid particles inside the boundary are mainly austenite structure rather than martensite structure. This phenomenon is due to the high alloying elements in the solid austenite during semi-solid temperature range [5]. Thus the martensite transformation is suppressed [20]. Hardness in the inner area is about 376 HV level, while hardness in the surface is about 726 HV level.

3.2. Strain hardening behavior inside the solid/liquid boundary

The compression tests were carried out in order to investigate the mechanical properties of T-T-550 specimen and clarify the evolution of deformation structure. As shown in Fig. 4 and Table 2, the test results were compared with that of CHT specimen. It can be observed that T-T-550 specimen demonstrates a long continuous strain hardening characteristics, in which compressive stress rises with compressive strain gradually. The material exhibits excellent mechanical properties (Compressive strength (R_{mc}) of 4680 MPa, Compression ratio of 53.2%) as compared with CHT specimen (R_{mc} of 3609 MPa, Compression ratio of 33.0%). The strain hardening ($d\sigma/d\varepsilon$ vs. compressive strain) behavior indicates T-T-550 specimen demonstrates multiple strain hardening. In particular, four different deformation stages can be distinguished for T-T-550 specimen. During the first stage (Stage A: 0–0.07), the strain hardening rate fluctuates sharply, which indicates the stabilization of the initial deformation. With further stress, the strain hardening rate increases gradually (Stage B: 0.07–0.27). Over a strain amount of 0.27, the strain hardening rate remains almost constant in a long deformation range (Stage C: 0.27–0.51). Finally, the strain hardening rate decreases drastically until failure of the specimen (Stage D: 0.51–). As for CHT specimen, three-stage strain hardening behavior can be observed without an increment in strain hardening rate after the initial stabilization.

In order to fully understand the multistage strain hardening behavior of T-T-550 specimen, microhardness evolution were investigated with the compression tests interrupted at predetermined strains (0.05, 0.1, 0.25, 0.50). The test results for different strains (0.05, 0.1, 0.25, 0.50) were 376.1 HV, 465.9 HV, 556.7 HV and 753.4 HV, respectively. It can be observed that microhardness increases with the strain, which corresponds to the continuous strain hardening characteristics.

T-T-550 specimen inside the solid/liquid boundary shows low yield strength, while shows high compressive strength and compression ratio. Excellent strain hardening behavior can be obtained for 9Cr18 steel after thixoforging and heat treatment. Thus the deformation mechanism for high strength and ductility of T-T-550