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ARTICLE

Experimental Study and Thermodynamic Analysis of Inclusion Precipitation during Solidification in 12% Cr Alloy

Yu Hansong, Li Jianguo

Shanghai Jiaotong University, Shanghai 200240, China

Abstract: Iron based 12%Cr alloy (mass fraction) is usually used for advanced electric rotors shaft which require very good mechanical property and fine inclusions, as the large-sized inclusions in the alloy can cause performance deterioration. In this work, quenching process was adopted after slow cooling experiment in order to retain the inclusions precipitated during melt cooling and solidification of 12% Cr alloy. The morphology and the composition of the inclusions were detected by scanning electronic microscopy (SEM) and energy dispersive spectrometry (EDS), respectively. The results show that besides various shapes of oxide, typical TiN inclusion is found in the sample. However, there is no Ti addition in metallurgical process. Thermodynamic calculation was executed to interpret the inclusion precipitation during solidification and the calculation results indicate that TiN inclusion precipitates at the terminal stage of solidification. Even a trace amount of Ti drawn from raw materials would induce the precipitation of TiN, which would grow into large size and be harmful to alloy property, while the formation of Al_2O_3 inclusion occurs in liquid alloy. Suggestions for the control of Ti and O are given based on the calculation results, which would be helpful to the production of 12%Cr alloy. The precipitation potential of Ti_2O_3 inclusion in the experiment has also been discussed by studying the competition of element concentration between Ti and Al.

Key words: inclusion precipitation; solidification; 12% Cr alloy; thermodynamic calculation

12% Cr alloy is used for manufacturing advanced electric rotor shafts which work under severe working condition (high temperature, steam corrosion, energy radiation, etc.) and require excellent performances on high-temperature strength, creep resistance, impact behavior, and so on[1]. Since inclusions reserved in the product have significant effect on properties and can cause kinds of problems such as strength weakening, fatigue failure, impaction degradation, and so forth[2], it is worthwhile to pay attention to the formation of inclusions in 12% Cr alloy, especially the inclusions formed during solidification, as inclusions which appear and grow up in this period are maintained in the final industrial production, not like primary inclusions which are mainly wiped out.

Many previous studies have been carried out to investigate inclusions during solidification by combining with thermodynamic analysis via kinds of materials [3-5]. However, there is few discussion about the formation of inclusions during solidification for 12% Cr alloy. Research works related to 12% Cr alloy mainly focused on the microstructure evolution [6,7], or carbonitrides precipitation like M*X* (M = metal, *X*=C, N) and Cr-based carbides ($Cr_{23}C_6$, Cr_7C_3) during heat treatment process [8,9]. But the control for inclusion is also one of the fundamental requirements in the production considering its industrial applicability. Therefore, more interest should be paid to the inclusion formation in 12% Cr alloy.

In the present study, a special procedure quenching was initiated after slow cooling experiment to reserve the inclusions formed during solidification in 12% Cr alloy, because inclusions observed in conventional solidification process can be controversial on whether they precipitate during melt cooling and solidification, or even in solid phase cooling period. The morphology and composition of inclusions were identified by SEM and EDS, respectively.

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Corresponding author: Li Jianguo, Professor, School of Materials Science and Engineering, Shanghai Jiaotong University, Shanghai 200240, P. R. China, Tel: 0086-21-54744119, E-mail: lijg@sjtu.edu.cn

Meanwhile, thermodynamic calculation was employed on types of inclusions which were displayed in the experiment so as to understand their formation. Based on thermodynamics a few suggestions for the composition controlling are given in order to improve the manufacture of 12% Cr alloy.

1 Experiment Procedure

The raw 12% Cr alloy with dimension of 75 mm in length and 25 mm in diameter was prepared. Then it was put into a high purity quartz crucible and placed in an induction furnace which was filled with argon atmosphere to 0.05 Pa gas pressure after evacuating by a mechanical pump and a molecular pump. The alloy was heated to 1873 K and held for 5 min in order to stabilize the melt. Subsequently it was cooled down at a cooling rate of 0.05 K/s by gradually turning down the power. Under such a slow cooling rate, the formation of non-metallic inclusion could be regarded in a quasi-equilibrium state. Finally when the temperature reached 1688 K, the solidus temperature of 12% Cr alloy, which was calculated by the software JMat-Pro, the sample was quenched in water to suppress the precipitation after solidification, so the inclusions which had formed from melt cooling and solidification were reserved. In the experiment, all the temperatures were recorded by an infrared temperature measurement system.

The chemical composition of experimental alloy is shown in Table 1. The content of carbon and sulfide were analyzed by carbon and sulfur analyzer while oxygen and nitrogen contents were analyzed via inert gas fusion-infrared absorptiometry. The component of other metallic elements such as Cr, Al, and Ti were analyzed by an inductively coupled plasma-atomic emission spectrometry (ICP). Though no titanium was added during metallurgical process, a trace amount of titanium were detected in the alloy. It is speculated that such few titanium drawn from scrap iron were the primary charge of raw material.

In order to observe the morphology and detect the composition of inclusions reserved in the alloy, preparation was as follows: firstly, the sample was cross-section cut, mechanically ground with silicon carbide emery paper down to 2000 grit, and then polished with diamond pastes down to 1 μm; subsequently it was ultrasonically rinsed in distilled water. After all these above, the examination on the sample was executed via a scanning electron microscopy (SEM, Jeol JSM-7600F) equipped with an energy dispersive spectrometry (EDS).

2 Results and Discussion

2.1 Precipitation of TiN inclusion in the experiment

Experimental results in Fig.1 show the morphology of TiN inclusion, which has a typical rectangular shape reported in references [10,11]. The size of TiN observed is about 2~3 μm and Fig.1b gives a clear magnification of it. EDS detection shows that the inclusion content (mass fraction) is: Ti 32.42%, N 18.88%, Fe 42.97% and Cr 5.73%, where Fe and Cr comes from the base material.

Thermodynamics of TiN in the alloy was calculated. Titanium has very strong affinity with nitrogen, and the reaction between Ti and N can be expressed by Eq. (1) [12]:

$$\mathrm{Ti}+\mathrm{N}=\mathrm{TiN}$$

$$\lg\frac{\alpha_{\mathrm{TiN}}}{\alpha_{\mathrm{Ti}}\cdot\alpha_{\mathrm{N}}}=\lg\frac{1}{f_{\mathrm{Ti}}[\%\mathrm{Ti}]f_{\mathrm{N}}[\%\mathrm{N}]}=17040/T-6.40 \quad (1)$$

where α_{Ti}, α_{N} are the activities of element Ti and N, respectively, and α_{TiN} is considered as 1 due to its low solubility. In the present study the activity of inclusion is unity (pure inclusion as the standard state). [%Ti], [%N] are the weight percentage of elements Ti and N, respectively. f_{Ti}, f_{N} are the elements activity coefficients which are determined by Wagner's formalism described in Eq. (2).

$$\lg f_i=\sum e_i^j[\%j] \quad (2)$$

where e_j^i is the first-order interaction parameters of element j to i, and [%j] is mass percent of element j. Their values are obtained from literatures available and listed in Table 2. The liquidus temperature and solidus temperature of 12% Cr alloy are calculated to be 1773 K and 1688 K by the software JMat-Pro, respectively.

Fig.2 shows the stability diagram of TiN precipitation during solidification for 12% Cr alloy. The curves were calculated according to Eq.(1) and lever rule which was used to describe solutes segregation during solidification in equilibrium state. Before calculation it was assumed that Ti consumption occurred only if TiN precipitation took place. The black point in Fig.2 represents the actual concentration of

Table 1 Chemical composition of 12% Cr alloy (wt%)

C	Si	Mn	P	S	Cr	Al	Ti	O	N
0.12	0.11	0.43	0.01	0.0086	10.53	0.01	0.005	0.005	0.054

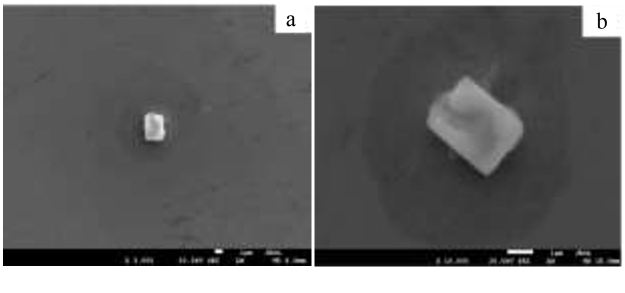

Fig.1 Typical TiN inclusion observed in the sample