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ARTICLE

Effect of Excess Fe_2O_3 on Microstructure and Tensile Properties of Micro-nano Structure 316L Austenitic Stainless Steels Prepared by Aluminothermic Method

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Abstract: The regulation of structure and composition is very important for obtaining stainless steel with good comprehensive properties. Fe_2O_3 powers with different excess fraction were added in reaction powders to make complete aluminothermic reaction and reduce the content of Al in 316L ASSs. The effects of excess Fe_2O_3 in reactions on microstructure and tensile properties of micro-nano structure of 316L austenitic stainless steels were analyzed. The results show that with the increase of Fe_2O_3 excess fraction, the volume fraction of ferrite phase reduces significantly while the volume fraction of nanocrystalline increases from 87.4% to 93.4% and the average grain size decreases from 32 nm to 22 nm. The comparative analysis shows that the Fe_2O_3 excess fraction of 5.0 % is the best processing parameter for the 316L ASSs preparation by the aluminothermic method. For the single phase austenitic steel prepared under this condition, the tensile strength is 573.92 MPa, the yield strength is 340.12 MPa, and the elongation is 4.68%.

Key words: aluminothermic reaction; micro-nano structure; 316L austenitic stainless steel; tensile property

316L austenitic stainless steels (ASSs) have been widely used in petrochemical, marine, paper and other industries due to their good resistance to high temperature, acid and chlorine corrosion, excellent processing and welding performance, etc[1-3]. However, their applications in more fields are restricted due to their lower strength and hardness[4,5]. Although nano-structures can greatly improve the yield and tensile strength, the plasticity is reduced[6,7]. In order to balance the strength and toughness, the micro-nano structure has been concerned by many researchers[8,11]. Ma[12] and Wang et al[13] proposed a method to optimize the plasticity of nanocrystalline materials, and pointed out that non-uniform structure (nanocrystalline+ultrafine or microcrystalline) is an effective measure. The hardening achieved by grain refinement is coupled with the ductility afforded by incorporation of coarse grains, which can achieve a combination of strength, ductility and toughness that was previously impossible. Han et al[14] pointed that a yield strength of 700 MPa was obtained in the fully cryomilled 5083 Al alloy, which compared favorably with the yield strength of conventional 5083 (145 MPa). As the volume fraction of coarse grains increased, strength decreased slightly, but ductility increased. That is, micro-nano structures can be taken into account to balance the strength and plasticity of stainless steel. Subsequently, the key solution is how to obtain the micro-nano structure.

Currently, there are two main ways to obtain a micro-nano structure. One is “small to large”, that is to prepare regular bulk materials by the following process: mixing the nano- and micro-powders, followed by ball milling, sintering, and hot extruding. The advantages of this approach are that the volume fractions of the micro-nano crystalline phases can be accurately controlled, and the mechanical properties of alloys can be further adjusted[15-17]. However, it is easy to introduce defects, impurities and large internal stresses with mixing the particles. The other is “big to small”, which refers to the large

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plastic deformation of bulk samples (such as low temperature cold rolling[18], equal channel angular pressing[19,20], high pressure torsion[21,22], plane strain compression[23]) to obtain metastable nano-structures, and then a multiphase structure is obtained through a heat treatment process since some nanocrystals preferentially grow to microcrystals[24]. However, this method is not suitable for most materials, and the size of the prepared materials is limited.

The aluminothermic reaction is a relatively novel method which is the reaction of high purity materials at low temperatures in an inert atmosphere. Through controlling the melt cooling process and extending the melt purification and solidification time, some crystal nuclei precipitate in the early stage, and then grow up as microcrystalline phase. The molten microcrystals provide a lot of nucleation sites for the nanocrystalline phase in the later stage. Thus, materials with micro-nano structures can be obtained[25].

However, our previous study[26] has found that some aluminum elements do not react completely and remains in steel. Al residue leads to ferrite (α) formation and inevitably reduces the percentage of other elements in the alloy. Fortunately, the addition of excess Fe_2O_3 can complete the Al reaction.

In present research, the excess fraction Fe_2O_3 will be added in the reaction powders to carryout completely aluminothermic reaction and reduce the content of Al in the 316L ASSs. The effects of excess fraction Fe_2O_3 on the microstructure and tensile properties of micro-nano structure 316L ASSs prepared by aluminothermic method will be also analyzed in detail.

1 Experiment

According to the chemical composition of the 316 ASS designed in this experiment (Table 1) and the chemical reaction of aluminothermic reaction (Eq.(1)):

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

the mass fraction of the standard reaction materials is obtained, as shown in Table 2. On the basis of standard proportion, Fe_2O_3 was added in excess fractions of 2.5%, 5.0% and 7.5% to obtain a series of alloy reaction raw materials. The particle size of raw materials is no more than 74 μm (200 mesh).

The powders were weighted and mixed with corundum ball at a ratio of 1:2 and then milled in QM-BP planetary ball mill for 8 h at a speed of 100 r/min. Then the mixed reactant powers were placed in the mold and pressed into compacts with a diameter of 85 mm and a height of 30 mm at a pressure of 35 MPa. A lamellar igniter was put on the surface of pressed powders, and then the tube with the powders was placed in a reactor, which is given in detail elsewhere[26]. Firstly, the reactor was purged with argon gas at room temperature and then heated to 180 °C. Secondly, it was filled with argon at 5 MPa and heated again to 250 °C. At this point, the lamellar igniter began to work and release exothermic heat that resulted in the aluminothermic reaction. Finally, the reaction completed in a few seconds, and the products were cooled to room temperature in argon before being taken out from the reactor and the Al_2O_3 layers were manually removed. Thus, the 316L ASSs were prepared by aluminothermic method with excess fractions Fe_2O_3 of 0% (without excess), 2.5%, 5.0% and 7.5%.

Table 1 Chemical composition of 316L ASSs (wt%)

C	Si	Mn	Cr	Ni	Mo	S	P	Fe
0.03	1.0	2.0	17	14	2	≤0.03	≤0.035	Bal.

Table 2 Standard proportion of 316L ASSs reaction raw materials (wt%)

Fe_2O_3	Al	Cr	Mn	Si	Ni	Mo	C
57.7	19.5	10.75	1.3	0.63	8.8	1.3	0.02

The microstructures were analyzed by a D/Max-2400 X-ray diffraction (XRD) with Cu Kα radiation and a JSM-6700F field emission scanning electron microscope (SEM). The transmission electron microscope (TEM) and selected area electron diffraction (SAED) observation was conducted by a JSM-2010 TEM operated at 200 kV. The TEM specimens were prepared by mechanical grinding to a thickness of about 40 μm, and then the disk with 3 mm in diameter was dimpled on both sides using a twin-jet electro-polishing. A solution of 5 vol% perchloric acid in ethanol was served as the electrolyte.

Tension measurements were performed at room temperature according to ASTM E8 standard by a Shimadzu AT10t testing machine at a crosshead speed of 0.2 mm·min^{-1}. The specimens for tensile and compression tests were cut from the 316L ASSs through the wire cutting method. The schematic diagram of tensile specimen is shown in Fig.1. Due to the small size of the tensile sample, extensometers cannot be added; therefore the elongation cannot be obtained directly through tensile curves. Here, the elongation (δ) was calculated according to the following formula:

$$\delta = (L_1 - L_0) / L_0 \times 100\% \quad (2)$$

where L_0 and L_1 are the gauge lengths before and after tension measurement, respectively.

2 Experimental Results

Fig.2 shows XRD patterns of the 316 ASSs prepared by aluminothermic method with different excess fractions of Fe_2O_3. It can be seen that the main phase of all samples is γ phase,

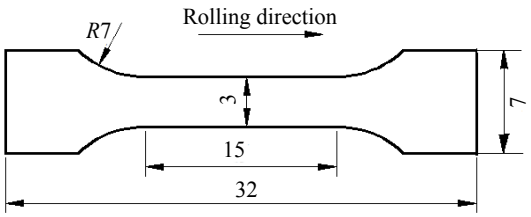

Fig.1 Schematic diagram of tensile specimen

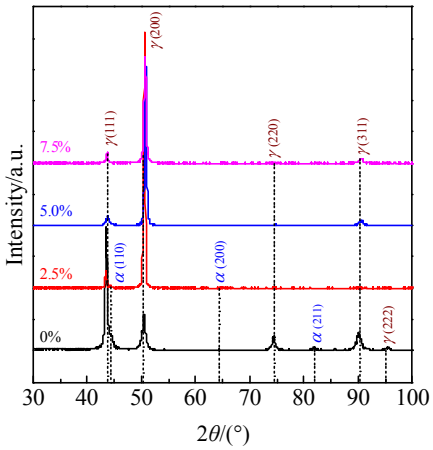

Fig.2 XRD patterns of 316 ASSs prepared with different excess fractions of Fe_2O_3

and the diffraction peaks of α phase appear in samples prepared without or with less excess Fe_2O_3. But with the increase of Fe_2O_3 excess fraction, the strongest diffraction peak of γ phase is converted from the theoretical γ (111) peak to the γ (200) peak, and the diffraction peaks of α phase disappear gradually, which preliminarily shows that the structure of 316 ASSs prepared by aluminothermic method can be controlled by adjusting the excess fraction of Fe_2O_3. Furthermore, the grain size of γ nanocrystalline in the alloys prepared with excess fraction Fe_2O_3 of 0%, 2.5%, 5.0% and 7.5% can be calculated by MDI Jade 5.0 software, and the values are 27, 34, 23 and 22 nm, respectively.

SEM micrographs of the 316 ASSs prepared with excess fraction Fe_2O_3 of 0%, 2.5%, 5.0%, 7.5% are shown in Fig.3 and EDS results of different phase regions are listed in Table 3. The samples prepared without or with less excess Fe_2O_3 are composed of the white matrix and the black phase. According

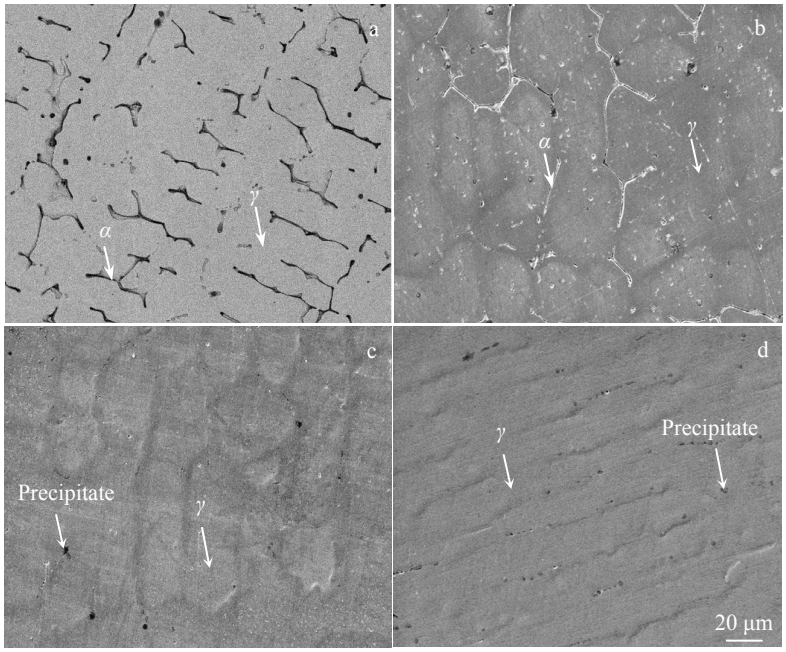

Fig.3 SEM micrographs of 316 ASSs prepared with excess fraction Fe_2O_3 of 0% (a), 2.5% (b), 5.0% (c), and 7.5% (d)

Table 3 EDS and Image J software statistical results of different phase regions in 316L ASSs

Excess fraction of Fe_2O_3/%	Phase	Composition/wt%							Volume fraction/%
		Fe	Cr	Ni	Mo	Si	Mn	Al	
0	γ	65.68	13.87	15.22	1.13	1.39	0.80	1.91	90.5
	α	63.78	16.78	12.79	2.27	1.36	0.57	2.45	9.5
2.5	γ	64.13	14.42	15.71	2.15	1.08	0.79	1.72	96.5
	α	63.14	17.10	9.57	5.29	2.21	0.60	2.11	3.5
5.0	γ	60.45	15.46	15.27	2.99	2.56	1.98	1.29	99.2
	Precipitate	42.52	19.28	11.30	20.43	3.60	1.85	1.02	0.8
7.5	γ	66.57	12.94	14.43	2.88	1.43	0.75	0.99	96.8
	Precipitate	4.70	14.73	1.20	53.13	0.24	25.62	0.38	3.2

to the EDS results, the white matrix is γ phase and the black is α phase. The volume fraction of different phase regions was statistically analyzed by Image J software, as listed in Table 3, according to five SEM micrographs of each alloy. For the alloy prepared with 2.5% excess fraction Fe_2O_3, the volume fractions of γ and α phases are 96.5% and 3.5%, respectively. Compared to the 316L ASSs prepared with the standard ratio, the volume fraction of α phase decreases from 9.5% to 3.5% and the Al content also decreases from 2.45% to 2.11%. However, when the excess fraction of Fe_2O_3 are 5.0% and 7.5%, there are only matrix phase and precipitates, and the Al content further reduces. Among them, the volume fraction of precipitates in the alloy with 7.5% excess fraction Fe_2O_3 increases to 3.2%, and in the γ phase, the Al content reduces to 0.99%. The content of Cr (another important alloying element) also drops sharply to 12.94%, which is beyond the normal range of Cr content in 316L austenitic stainless steel. Furthermore, the content of Mo and Cr in the precipitates is higher than that of the matrix, which confirms that the precipitates are FeCrMo intermetallic compounds.

The typical bright and dark field TEM micrographs and corresponding SAED pattern of the 316L ASSs prepared with 2.5% excess fraction Fe_2O_3 are given in Fig.4. The small black spots in bright field TEM image, which correspond to the small bright spots in dark field, are nanocrystalline grains. The SAED pattern (Fig.4b) shows that all diffraction rings come from γ grain nanocrystallines, and there are free of α grains. According to the statistics of grain size in several dark field images by Image J software, the volume fraction of nano-crystals and microcrystals are 87.4% and 12.6%, respectively. The grain size distribution is shown in Fig.4d. The average grain sizes of nanocrystals and microcrystals are 32 and 215 nm. Therefore, after adding an excess of 2.5% Fe_2O_3, the 316L ASSs still maintains the micro-nano structure.

According to TEM images in Fig.5, the 316L ASSs prepared with 5.0% excess fraction Fe_2O_3 is still composed of a large number of nanocrystals and a small number of microcrystals. From the SAED pattern (Fig.5b), there is still only the diffraction ring of the fcc structure, which further confirms that only γ structure exists without α structure. The average grain size is obtained by counting the grain size of five dark field images by software, as shown in Fig.5d; the average grain size of nano-crystals is about 29 nm, and the volume fraction is 90.1%. For microcrystals, the grain size is 134 nm, and the

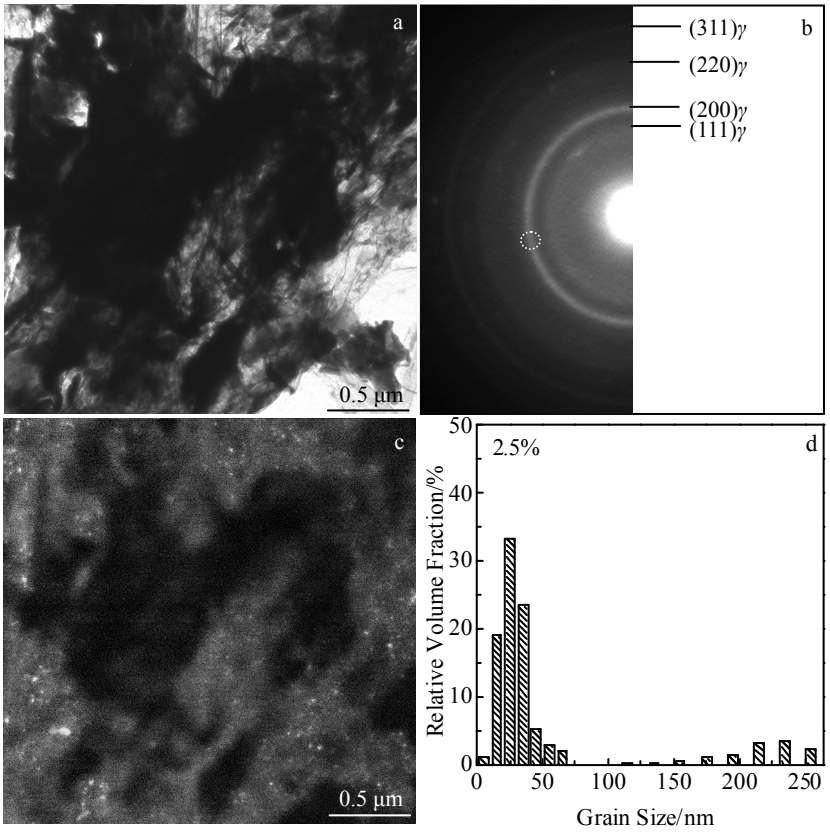

Fig.4 TEM analysis results of 316L ASSs prepared with excess fraction Fe_2O_3 of 2.5%: (a) bright-field image, (b) corresponding SAED pattern, (c) dark-field image and (d) histogram of grain size distribution determined from dark field images (dotted circle in SAED pattern indicates the operating spots of the dark-field image)

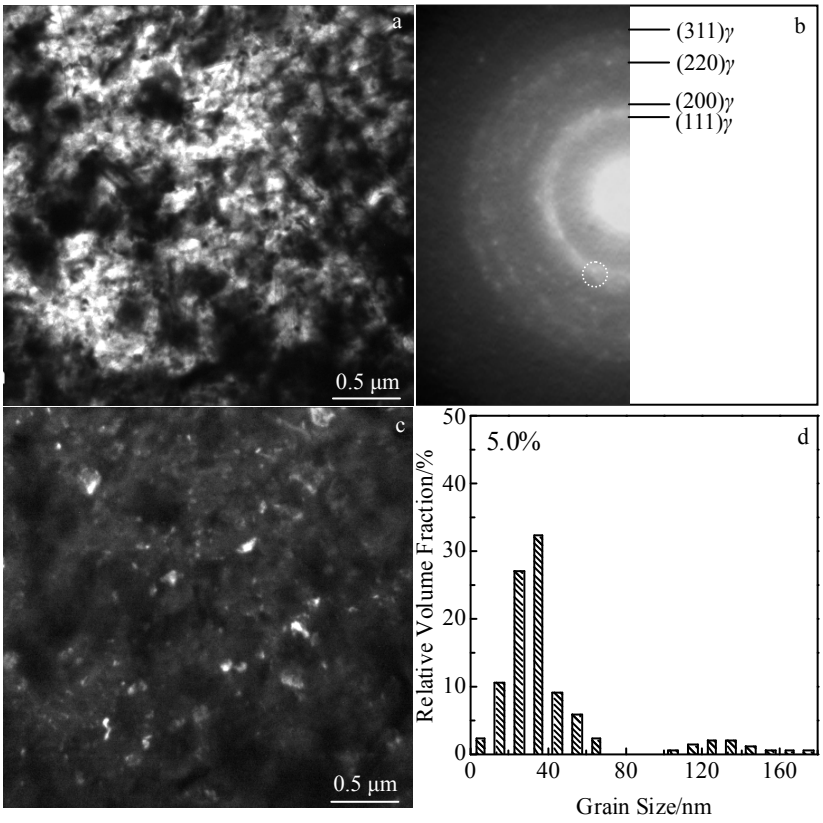

Fig.5 TEM analysis results of 316L ASSs prepared with excess fraction Fe_2O_3 of 5.0%: (a) bright-field image, (b) corresponding SAED pattern, (c) dark-field image and (d) histogram of grain size distribution determined from dark field images (dotted circle in SAED pattern indicates the operating spots of the dark-field image)

volume fraction is 8.9%. In view of the above characterization of 316L ASSs obtained by aluminothermic reaction with 5.0% excess fraction Fe_2O_3, it is known that further increasing the excess fraction of Fe_2O_3 can actually reduce the Al content in stainless steel, thus eliminating α phase successfully. Furthermore, the stainless steel still maintains the micro-nano structure.

Fig.6 shows TEM results of the 316L ASSs prepared with 7.5% excess fraction Fe_2O_3. The grey areas in bright field image and the bright spot areas in the dark-field image are nanocrystalline grains, which are confirmed by the SAED pattern (Fig.6b). While the white areas in the bright field image and the black areas in the dark field image are microcrystal distribution regions. Namely, the microstructure of this alloy is still composed of microcrystals and nanocrystals. The statistically obtained nanocrystals have an average size of 22 nm and a volume fraction of 93.4%; the average size of microcrystals is 156 nm, and the volume fraction is 6.6%.

Fig.7 presents the tensile stress-strain curves of the 316 ASSs with different excess fraction of Fe_2O_3. It can be seen that the tensile process of the 316 ASSs has experienced the elastic strain phase, the yield phase and the plastic deformation phase, and it has obvious plastic material characteristics. Compared with the 316L ASSs prepared with 0% excess fraction Fe_2O_3, the yield strength of the 316L ASSs prepared with 2.5% excess fraction Fe_2O_3 decreases significantly, the tensile strength decreases from 563.18 MPa to 331.2 MPa, and the yield strength decreases from 271.83 MPa to 153.13 MPa. When the excess fraction of Fe_2O_3 is 5.0%, the strength of the alloy increases significantly, the tensile strength is 573.92 MPa and yield strength is 340.12 MPa. For the alloy prepared with 7.5% excess fraction Fe_2O_3, the tensile strength decreases to 374.44 MPa and the yield strength decreases to 175.62 MPa. However, the elongation decreases with the increase of excess fraction of Fe_2O_3. For the single phase 316L ASSs prepared with 5.0% excess fraction Fe_2O_3, the elongation is 4.68%.

Fig.8 shows the SEM morphologies of tensile fracture of 316L ASSs prepared with excess fraction Fe_2O_3 of 5.0%, which includes both stream patterns representing brittle fracture (Fig.8a) and dimpled fractures (Fig.8b) representing ductile fracture, and it indicates that dimpled fracture and brittle fracture exist simultaneously. The average diameter of dimples is about 1 μm, and some coarse dimples contain particles, which should be FeCrMo intermetallic compound particles. In the process of stretching, stress concentration tends to occur around the particles in the second phase to form

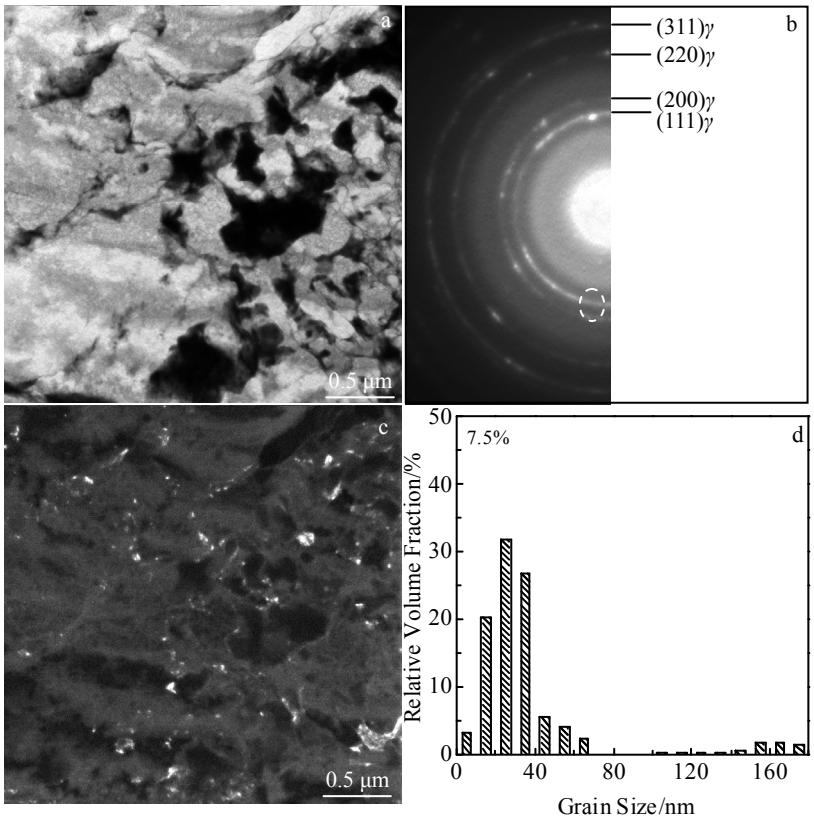

Fig.6 TEM analysis results of 316L ASSs prepared with excess fraction Fe_2O_3 of 7.5%: (a) bright-field image, (b) corresponding SAED pattern, (c) dark-field image and (d) histogram of grain size distribution determined from dark field images (dotted circle in SAED pattern indicates the operating spots of the dark-field image)

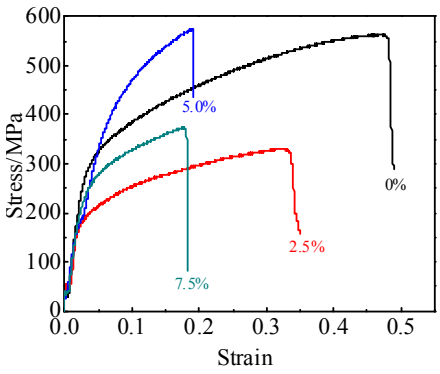

Fig.7 Tensile stress-strain curves of 316L ASSs prepared with different excess fractions of Fe_2O_3

micro-holes. With the continuous increase of stress, micro-holes gradually increase and grow, and finally connect to each other, forming fracture dimples around the particles.

3 Discussions

3.1 Effect of excess Fe_2O_3 on phase evolution

Al element is one of the elements promoting ferrite formation,

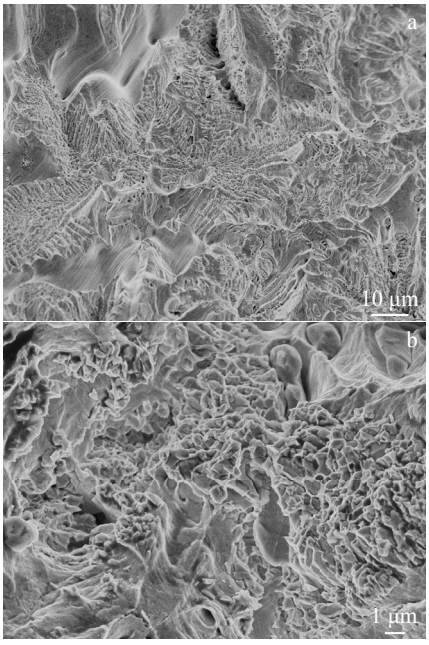

Fig.8 SEM morphologies of tensile fracture of 316L ASSs prepared with excess fraction Fe_2O_3 of 5.0%: (a) stream patterns and (b) dimpled fractures

it is very important to control the Al content in the formation of single-phase austenite steel. For steel alloys, the Cr equivalent ($Cr_{eq.}$) is usually used as the measurement index of ferritization, and the formula is: $Cr_{eq.}$=[Cr]+1.5[Mo]+ 1.5[Ti]+ 1.75[Nb]+1.5[Si]+5.5[Al]+0.75[W]. It can be seen that the ability of Al to form α is 5.5 times larger than that of Cr, that is, a small amount of Al will greatly expand the α region. Therefore, when the 316L ASSs synthesized without excess fraction Fe_2O_3 contains Al content of about 2.45% in α phase, the volume fraction of α phase will reach about 9.5%.

In this research, Al content and the volume fraction of α phase can be effectively controlled by adding different excess fractions of Fe_2O_3 in aluminothermic reaction. When 2.5% excess fraction Fe_2O_3 is added, the contact probability of Fe_2O_3 and Al powder increases during the reaction, so they can fully react, which contributes to decreasing the Al content in the matrix. However, the Al powder still cannot react sufficiently, and there are still 2.11% Al and 3.5% α phase. When the excess fraction of Fe_2O_3 rises up to 5.0%, the Al powder can further react and the Al content reduces to about 1.29%. There is no α phase, but only a small amount of FeCrMo intermetallic compounds in the alloy. However, when the Fe_2O_3 powder continues to increase to an excess fraction of 7.5%, the Al content reduces to 0.99% in γ phase, and the FeCrMo intermetallic compound significantly increases, which will seriously reduce the corrosion resistance of stainless steel due to the precipitation of alloying elements[27-30]. In addition, the Cr content significantly reduces, which deviates from the composition standard of 316L ASS. Therefore, based on the characterization and the discussion above, it can be concluded that the Fe_2O_3 excess fraction of 5.0% is the best for the 316L ASSs preparation by aluminothermic method.

3.2 Effect of excess Fe_2O_3 on average grain sizes

With an increase of Fe_2O_3excess fraction, the average grain size of the 316L ASSs reduces from 32 nm to 29 nm, and then to 22 nm. The main cause is that during sample preparation without or with less excess Fe_2O_3, some Al remains, resulting in insufficient aluminothermic reaction of raw materials. Thus the total heat released by the reaction is lower than that of the full aluminum thermal reaction when the excess ratio of Fe_2O_3 is larger, and the maximum temperature of the melting pool is also relatively low. Namely, when the aluminum thermal reaction is sufficient, the maximum temperature of the molten pool will be higher. However, the higher the molten pool temperature, the greater the temperature decline rate in the initial stage under the same cooling condition. With the increase of cooling speed, the undercooling degree also increases. In the case of larger undercooling degree, the nucleation rate increases faster than the rate of crystal growth, so finer grains are obtained [31]. Moreover, the FeCrMo intermetallic compound increases with increasing the Fe_2O_3, which also relates to the lower temperature, because it is difficult for the FeCrMo compound to dissolve in the matrix under the condition of lower temperature and shorter cooling time[32,33].

3.3 Effect of excess Fe_2O_3 on tensile properties

Based on the tensile test results, it can be seen that the tensile properties do not change linearly with excess fraction of Fe_2O_3. For the alloy prepared with excess fraction Fe_2O_3 of 2.5%, its ferritization degree decreases compared with the alloy without excess Fe_2O_3, and its strength also decreases correspondingly. When the excess fraction of Fe_2O_3 increases to 5.0%, the strength reaches the maximum value. Because the alloy is a single-phase austenitic stainless steel alloy, there is a small amount of FeCrMo intermetallic compounds which are also the strengthening phase dispersed in the matrix and hinder the movement of the dislocations. When the dislocation lines in the matrix move to the vicinity of the strengthening phase, they will be obstructed or bent. As the applied stress increases, the dislocation lines are stretched and curved into a dislocation loop which is continuously intersected, making it difficult for the dislocation to move in the crystal and increasing the strength significantly. However, it should be noted that the strengthening phase of FeCrMo intermetallic compound adversely affects the corrosion resistance of stainless steel. Therefore, it is expected to be eliminated by the subsequent solution treatment.

4 Conclusions

1) Excess Fe_2O_3 added in the reaction powders can make the aluminothermic reaction complete and reduce the content of Al in the 316L ASSs.

2) With the increase of Fe_2O_3 excess fraction, the volume fraction of α phase reduces significantly, while the volume fraction of nanocrystals increases from 87.4% to 93.4% and the average grain size decreases from 32 nm to 22 nm.

3) The comparative analysis shows that the Fe_2O_3 excess fraction of 5.0% is the best process parameter for the 316L ASSs preparation by aluminothermic method. For the single phase austenitic steel prepared with excess fraction Fe_2O_3 of 5.0%, the tensile strength is 573.92 MPa, the yield strength is 340.12 MPa, and the elongation is 4.68%.

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过量 Fe_2O_3 对铝热法制备微纳结构 316L 奥氏体不锈钢组织和拉伸性能的影响

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摘　要：不锈钢结构和成分的调节对获得良好综合性能至关重要。为了使铝热反应完全、降低 316L 奥氏体不锈钢（ASSs）中残余 Al 的含量，在反应粉体中加入了不同过量分数的 Fe_2O_3 粉末，详细分析了过量 Fe_2O_3 对 316L ASSs 微纳米组织和拉伸性能的影响。结果表明，随着 Fe_2O_3 过量分数的增加，铁素体相的体积分数显著降低，而纳米晶的体积分数由 87.4%提高到了 93.4%，平均晶粒尺寸由 32 nm 降低到了 22 nm。对比分析表明，铝热法制备 316L ASSs 的最佳工艺参数为添加过量分数 5.0%的 Fe_2O_3，在该条件下制备的单相奥氏体钢，其抗拉强度为 573.92 MPa，屈服强度为 340.12 MPa，延伸率为 4.68%。

关键词：铝热反应；微纳结构；316L 奥氏体不锈钢；拉伸性能

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