Effect of Zirconium Oxide Addition on Mechanical Properties in Ultrafine Grained Ferritic Stainless Steels^{*1}

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Zirconium (Zr) and 12 mass% chromium containing ferritic stainless steels with Zr oxide dispersoids have been developed. In this study, the relationship between the process conditions and the metallurgical and mechanical properties were investigated. 12Cr-1Zr steel was consolidated with a grain size of about 1 μ m by extrusion process of powder without mechanical alloying (MA). On the other hand, 12Cr-1Zr steels with MA process were shown to have a grain size of only about 0.36 μ m or less. It was suggested that Zr atoms strongly reacts with gaseous impurities such as oxygen and carbon that were entrapped during MA. Based on TEM observations, Zr oxide and carbide preferentially precipitated on the grain boundaries which pin the grain boundary migration during the extrusion process. The developed steels follow the Hall-Petch relation and the slope is nearly identical to that of pure iron. Charpy impact values, at room temperature, of over 3 MJ/m2 up to tensile strength of 1500 MPa were obtained which is three times higher than those of conventional ferritic stainless steels or PH-stainless steels. [doi:10.2320/matertrans.MRA2008036]

(Received January 28, 2008; Accepted April 1, 2008; Published May 14, 2008)

Keywords: mechanical alloying, ferritic stainless steel, zirconium oxide, grain refinement, tensile strength, Charpy impact test

1. Introduction

Grain refinement is one of the strengthening methods for metallurgical materials that gives a diluting effect of impurities at grain boundaries, relaxation of stress concentration at triple points and homogeneous properties so that higher strength could be obtained without losing ductility.¹⁾ The past few years have seen a steady progress on research activities in nano-crystallization technique, and micro-structurally controlled materials with several times higher strength than conventional materials have been manufactured.²⁾ Therefore, the manufacturing process is significant for environmentconscious materials to achieve a perfect balance between high strength and ductility without changing the chemical composition.

In the steel industry, recent developments have been made on ultra-fine crystalline steels by introducing further large reduction and strain to the conventional TMCP (Thermo Mechanical Control Process) to refine the grain size down to $1 \mu m.^{3,4)}$ Other techniques, such as MA (Mechanical Alloying), ECAP (Equal Channel Angular Pressing) and ARB (Accumulative Roll Bonding) have also been developed for nano-scale ultra-fine microstructure.⁵⁾ Among these processes, MA can be easily adapted to mass production of ultra-fine crystalline powders. MA is a powder metallurgy process, which has already seen commercial implementations for plates, rods, pipe and blocks of ODS (Oxide Dispersion Strengthened) alloys. Therefore, MA can be applicable to a manufacturing method for small scale structural components with ultra-fine crystalline materials.

Grain growth during sintering process is one of the key issues for powder metallurgy. It is possible to homogeneously distribute fine dispersoids such as oxide by MA to prevent grain growth such that ultra-fine crystalline steels with Y_2O_3 , TiO_2 , Fe_3O_4 are formed.^{6–10)} It is also noted that unavoidable impurities such as oxygen (O) and carbon (C) are mixed into the powder from the atmosphere, balls, tank and fixtures, which exerts a harmful influence on the mechanical properties.

Zirconium (Zr) shows a great affinity for impurities such as O or C. It is also expected to form oxides or carbides, which act as a pinning dispersoid against grain boundary migration. Pinning effect of grain boundaries by Zr carbide was reported for austenitic stainless steels.^{11,12)} In this paper, the pinning effect of Zr oxide in ultra-fine crystalline ferritic stainless steels is discussed. MA method and hot extrusion are employed to prepare the specimen of ultra-fine crystalline ferritic stainless steel. Zr is added as Zr powder or Zr oxide powder during MA process. Effect of the amount of chemical composition and the extrusion temperature on the grain size and the mechanical properties are investigated by tensile and Charpy impact testing at room temperature.

2. Experimental Procedure

2.1 Sample preparation

The base metal of Iron (Fe) comprising of 12 mass%Chromium (Cr) was vacuum melted and gas atomized to make raw powder. The powder was size classified to less than $180 \,\mu\text{m}$. The chemical composition (representative value) of the raw powder is listed in Table 1. MA was performed by using an attrition mill shown in Fig. 1. The raw powder and balls were put into the tank and milled by the arms attached on the rotating shaft. The tank, ball and arm are made of JIS SUS304, JIS SUJ2 and JIS SKD11 respectively. There

Table 1 Chemical compositions of primary powder (mass%).

Powder	Fe	Cr	Si	Mn	Р	S	С	0	Ν
Fe-12Cr	Bal	12.4	< 0.01	< 0.01	0.003	0.001	0.002	0.07	0.002

^{*&}lt;sup>1</sup>This Paper was Originally Published in Japanese in J. Japan Inst. Metals 71 (2007) 284–290.

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Fig. 1 Schematic drawing of attrition-mill.

are inlet and outlet ducts for cooling water. An inert gas (Ar) was introduced into the tank via gas inlet on the top. As a pretreatment of the milling apparatus, raw powder of 1 kg and balls of 30 kg were placed into the tank and milled for 20 hours to coat the ball, tank and arm surface with the raw powder.

Zr powder of 98% purity and less than 45 μ m average grain size (Kojundo Chemical Laboratory Co, Ltd) and ZrO₂ powder were utilized as an additive. The raw powder and additive were ball milled first and put into the attrition mill to be mechanical alloyed (MAed). The weight ratio of the powder and balls was fixed to 1 (2 kg) : 15 (30 kg). MA time was set to 100 hours, after which the clear peaks of Zr and/or ZrO₂ disappeared in X-ray diffraction. Ar gas was fed at a flow rate of 30 mL/min into the tank during MA and the cooling water was circulated around the tank to maintain a temperature of less than 293 K.

MAed powders were size classified to less than $180 \,\mu\text{m}$ again and put into a can made of mild steel. The can was vacuumed down to 0.4 Pa for 2 hours at 673 K and sealed. Then, the can was heated to $1023 \sim 1123$ K for 2 hours and isostatically extruded with an extrusion ratio of $5.0 \sim 5.7$.

Table 2 summarizes the fabrication conditions of the specimens. GA, the gas atomized Fe-12Cr-1Zr extruded at 1073 K, was prepared as a reference for MA-2, which was MAed and extruded.

MA-1, MA-2 and MA-3 contain 1 mass% Zr additive during MA and extruded at 1023 K, 1073 K and 1123 K respectively to consider the effect of extrusion temperature both on grain size and mechanical properties.

MA-4, MA-5 and MA-6 contain 1 mass% Zr additive and 0.08, 0.39 and 0.77 mass% ZrO₂ additive during MA and extruded at 1073 K. In these specimens, the oxygen amount supplied via ZrO_2 powder are 0.02, 0.1 and 0.2 mass% respectively, and hence the amount of ZrO_2 or Oxygen both on grain size and mechanical properties will be discussed.

MA-7 and MA-8 contain 0.77 mass% ZrO_2 additive during MA and extruded at 1073 K and 1123 K. In these specimens, only ZrO_2 was added and will be compared with Zr added specimens.

Table 3 shows the chemical composition of all the specimens. The amount of oxygen and carbon entrapped during MA is less than 0.1 mass% and 0.07 mass% respectively. MA-1, 2, 3, 4, 5 and 6 contain enough Zr assuming all the carbon and oxygen were consumed to form ZrC and ZrO₂ stoichiometrically.

2.2 Microstructural observations

The microstructure was observed by a 300 kV transmission electron microscope (TEM). The disk specimen of 3 mm in diameter and 0.3 mm in thickness was cut from the extrusion, polished with water abrasive paper down to 0.15 mm in thickness and finally electro-chemically polished using a twin jet thinning with 10% perchloric acid (HClO₄) + 90% acetic acid (C₂H₄O₂). In this study, the grain size was measured by means of intercept method on the transverse plane of the extrusion.

An assumption was made to calculate the volume fraction of the dispersoid in Zr added steels, i.e. all the carbon and oxygen analyzed as shown in Table 3 reacted with Zr to form ZrC and ZrO₂ stoichiometrically. The densities of ZrO₂, ZrC and the steel were assumed to be 6.0, 6.7 and 7.8 g/cm³ respectively. The effect of the nitrogen entrapment during MA was ignored, since the nitrogen entrapment is less than 0.01 mass%. Furthermore, the grain size of the dispersoid was assumed to be the same if the extrusion temperature was the same. In case of MA-7 and MA-8, the volume fraction of ZrO₂ additive was considered unchanged during the process.

Steel	Primary Powder (Gas-atomaized)	Quantity of adde (ma	d powder by MA ss%)	MA time (h)	Extruding temperature	
	-	Zr	ZrO ₂	_	(K)	
GA	Fe-12Cr-1Zr	_	_	_	1073	
MA-1	Fe-12Cr	1.00	—	100	1023	
MA-2	Fe-12Cr	1.00	—	100	1073	
MA-3	Fe-12Cr	1.00	—	100	1123	
MA-4	Fe-12Cr	1.00	0.08	100	1073	
MA-5	Fe-12Cr	1.00	0.39	100	1073	
MA-6	Fe-12Cr	1.00	0.77	100	1073	
MA-7	Fe-12Cr	_	0.77	100	1073	
MA-8	Fe-12Cr	_	0.77	100	1123	

Table 2 Fabrication proceedure of the specimens.

Table 3 Chemical compositions of the specimens.

Steel	Chemical composition (mass%)									
	Fe	Cr	Zr	Si	Mn	Р	S	С	0	
GA	Bal	12.0	0.93	_	_			0.01	0.05	
MA-1	Bal	11.8	0.88	_	_			0.07	0.09	
MA-2	Bal	11.7	0.90	< 0.01	0.02	< 0.005	0.001	0.07	0.09	
MA-3	Consist of the equivalent of "MA-2" elements									
MA-4	Bal	11.8	0.96	_	_			0.07	0.13	
MA-5	Bal	11.9	1.20	_	_			0.04	0.20	
MA-6	Bal	11.7	1.40	_	_			0.04	0.32	
MA-7	Bal	11.7	0.49	0.05	0.01	< 0.005	< 0.001	0.05	0.36	
MA-8	Consist of the equivalent of "MA-7" elements									

2.3 Mechanical testing

Tensile and Charpy impact tests were carried out at room temperature using the specimens shown in Fig. 2. Both the specimens were cut along with longitudinal direction of the extrusion. The tensile specimen is a flat plate of 25 mm gage length with $4 \text{ mm} \times 3 \text{ mm}$ cross section and tested at the nominal strain rate of $3.3 \times 10^{-4} \text{ s}^{-1}$. Extensioneter was utilized to measure the stress-strain curve. Reduction of area was measured from the fracture specimen. Charpy impact test was performed using test stand with 300 J capacity for JIS V notch half size specimen with 5 mm thickness.



Fig. 2 Test pieces for a) tensile test and b) Charpy impact test.

3. Results and Discussions

3.1 Microstructure

Figure 3 shows the appearance of the extruded specimen of 900 mm long and 25 mm diameter including mild steel can. Net sample size is about 700 mm long and 20 mm diameter. TEM micrographs and the average grain size of the specimens are shown in Fig. 4. As a reference, the microstructure of powder metallurgical processed and hot extruded JIS SUS410L is compared. The average grain size of 410 L is around $20 \sim 30 \,\mu\text{m}$, which is similar to commercial steel. On the other hand, the average grain size of GA with 1 mass% Zr, just extruded without MA, is about 1 μm , hence the addition of Zr affects the grain refinement regardless of MA. Moroishi¹³⁾ reported the effect of Zr addition on the ferritic stainless steel JIS SUS430. It is concluded that the dispersoid of Zr carbide or nitride prevents the grain growth up to 1273 K.

In case of GA, the dispersoids of $50 \sim 100$ nm are observed, while much finer ones are formed in MAed specimens. MA-2 has a similar chemical composition as GA, however, the grain size is $0.36 \,\mu\text{m}$ and hence MA has a strong effect on the grain refinement.

It is considered that the sort of dispersoid and the precipitation site are really important for the pinning of the grain boundaries as well as the grain size. However, it is considered that both additives of Zr and ZrO_2 have been formed into pinning dispersoid, which contribute to suppress the grain growth by pinning.

Hidaka¹⁰⁾ reported that the pinning effect is more significant with soluble oxides (such as Al_2O_3 , TiO₂ and Y₂O₃) than with non-soluble oxides (such as SiO₂ and Cr₂O₃).



Fig. 3 Appearance of extruded specimens.



Fig. 4 Optical micrograph (SUS 410L) and TEM images (GA, MA-1, MA-2, MA-3, MA-4, MA-5, MA-6, MA-7 and MA-8) of the specimens. SUS 410L was produced by powder metallurgy process with hot-extrusion.

However, decomposition of ZrO_2 during MA and its reprecipitation during the extrusion process are not clear, so that the behavior of additive of Zr and ZrO_2 during the process has been discussed.

Figure 5 shows the elemental mapping around the precipitations in MA-1 observed by EELS (Electron Energy Loss Spectroscopy). The precipitation contains Zr, O and/or C which suggests it is Zr oxide or carbide. Furthermore, Zr oxide tends to be observed at the grain boundary. Even though MA-1 was extruded at the lowest temperature of 1023 K in this study, Zr behaved as a getter of O and C during MA. Furthermore, solutioning of the precipitates was not observed in MA-2 and MA-3 even though those specimens were extruded at a higher temperature of 1023~1123 K. However, the grain size gets larger for higher extrusion temperature.

In order to investigate the precipitation site, MA-1 was heat treated for grain coarsening at 1223 K for 2 hours, at which TAGG (Transformation Assisted Grain Growth) occurs via $\alpha \rightarrow \gamma$ transformation.¹⁰⁾ As seen in Fig. 6, small particles are located as net-like distribution with almost the same spacing as that of the as-extruded with a grain size of about 200 nm, hence the precipitation site of the dispersoid is suggested to be at the grain boundaries in MA-1. Therefore, the dispersoid was formed with Zr and O, C introduced during MA and precipitated preferentially at grain boundaries, hence significant pinning of grain boundary migration was observed in MA-1, 2, 3, 4, 5 and 6.

In the case of these materials, the pinning particles are considered to be mainly Zr oxide as well as carbide. Zener's equation is well known for pinning effects of dispersoid for grain boundary migration, by which the grain size is given as



Fig. 5 Elemental map (C, O, Cr and Zr) around the precipitations in MA-1; analyzed by EELS[area a) and b)].



Fig. 6 a) TEM images of MA-1 after annealing at 1223 K for 2 h. b) Enlarged photograph. Dotted lines indicate the traces of the dispersed particles.

a function of the size and the volume fraction of the dispersoid.¹⁴⁾ However, by Zener's equation, it could underestimate the number of dispersoid as pinning particles for grain boundary migration, since the particles on an arbitrarily selected plane are considered to be at grain boundaries. Therefore, several modified equations are proposed by Nishizawa or Doherty^{15,16)} as follows:

Zener's equation:
$$D = 4d/3f$$
, (1)

by Nishizawa: $D = 8d/3f^{2/3}$

Modified Zener's equation

and

Doherty's equation:
$$D = 2d/f^{1/2}$$
, (3)

where D: grain size, d: dispersoid size and f: volume fraction of dispersoid. Nishizawa modified the equation considering the number of pinning particles at grain boundaries. Doherty took into the consideration the segregation of the dispersoid at the grain boundaries. Furthermore, Doherty's equation was modified under the assumption that (1) the radius of grain boundary is half the average grain radius, and (2) half the amount of dispersoid is located at the grain boundaries, which Takano applied for the commercial stainless steels of JIS SUSXM7 and SUS410.^{17,18)} The size of dispersoid was measured with TEM for several fields of MA-1, 2 and 3, which was measured as $5 \sim 10$ nm, $5 \sim 15$ nm and $10 \sim 25$ nm, respectively. It was also assumed that the size of dispersoid is a function of the extrusion temperature. Figure 7 shows the relation between the volume fraction of the Zr oxide and carbide and the grain size of both experimental and estimation based on the above equations. Even though some of the plots agree with Nishizawa's equation, all of the plots including ZrO₂ additive MA-7 and 8 show the better agreement with Doherty's equation, which assuming the half of the dispersoid are at grain boundaries.

Figure 8 shows the relation between the grain size and the amount of Zr oxide in MA-2, 4, 5, 6 and 7 which were extruded at the same temperature of 1073 K. It is clearly suggested that the grain size is a function of the amount of ZrO₂, even though calculated under the assumption that (1) dispersoid is Zr oxide (ZrO₂), (2) all the oxygen entrapped during MA (see, Table 3) is consumed by Zr and forms ZrO₂



Fig. 7 Relationship between estimated and measured grain-size of the specimens.

in MA-2, 4, 5 and 6, since the amount of Zr in these specimens is more than enough considering the stoichiometry of ZrO_2 , and (3) the amount of ZrO_2 additive are maintained during the process in MA-4, 5, 6 and 7. MA-7 shows the same trend as those of MA-2, 4, 5 and 6, hence it is suggested that the pinning effect depends on the amount of ZrO_2 . Therefore, it can be said that the additive ZrO_2 once decomposes during MA and re-precipitates during the extrusion, so that detail observations are required in the future.

3.2 Mechanical properties

Figure 9 shows the stress-strain curves of extruded specimens at room temperature. It can be said that the extrusion temperature strongly affects the mechanical properties by comparing MA-1, 2 and 3. It is also suggested that the amount of Zr and oxygen increase the mechanical strength. This is because the grain size is significantly refined for lower extrusion temperature and higher Zr and O content, since the grain growth is suppressed with the dispersoid preferentially



Fig. 8 Effect of zirconium oxide as ZrO_2 concentration on grain size of the specimens extruded at 1073 K.



Fig. 9 Stress-Strain curves of the hot extruded specimens.

formed at grain boundaries. As seen in Fig. 10, Hall-Petch relation is formed in these specimens. Furthermore, the gradient and strength value agree with those of pure iron, hence the grain refinement seems to be the dominant mechanism.

However, the uniform elongation decreases for higher strength. Especially over 1300 MPa, no uniform elongation was observed and necking occurred just after exceeding the elastic region. Similar phenomenon is also reported for grain refined pure iron, aluminum alloys and titanium alloys.^{6,19,20)} Uniform elongation is related to the work hardening rate, $d\sigma_t/d\varepsilon_t$, which needs to be larger than $d\sigma_t$ for the stability of plastic deformation, where $d\sigma_t$ and $d\varepsilon_t$ are true stress and



Fig. 10 Relationship between 0.2% proof stress and inverse of square root of grain size.



Fig. 11 Effect of oxygen content on 0.2% proof stress of the bulks extruded at 1073 K.

strain. It is considered that the plastic instability (necking) shows up in the early stage of the deformation since the work hardening rate does not change much.²¹⁾ Therefore, less uniform elongation is thought to be the nature of the ultra-fine crystalline materials, rather than the soundness of the specimen such as voids or defects.

0.2% proof stress of MA-2, 4, 5, 6 and 7 is plotted as a function of oxygen content in Fig. 11. These specimens were extruded at the same temperature of 1073 K. It is obvious that the higher oxygen content gives higher proof stress. Oxygen entrapped during MA is considered to be consumed by the additive of Zr, which forms ZrO_2 during the extrusion in MA-2, 4, 5 and 6. However, in case of MA-7 with additive of ZrO₂, entrapped oxygen is surplus so that no additional ZrO_2 is formed since there is no free Zr.

Figure 12 shows Charpy impact value and tensile strength of the extruded specimen and commercial JIS SUS410 (quenched and tempered)²²⁾ and PH-stainless steel.²³⁾ The specimen with Zr additive (MA-1, 2, 3 and 4) shows higher



Fig. 12 Variation of Charpy impact value and tensile strength of the specimens.



Fig. 13 Relationship between Charpy impact value and reduction area of the specimens.

toughness over 3 MJ/m^2 than the commercial steels. Among them, MA-4 shows strength up to 1500 MPa and toughness over three times higher than that of commercial steels.

Generally, toughness can be estimated from the area under stress-strain curve, see Fig. 9. However, only ZrO₂ added MA-8 shows relatively lower tensile strength and toughness. Figure 13 shows the relation between Charpy impact value and reduction of area of the extruded specimens. In case of MA-1 and 4, a reduction in area of over 50% was achieved, even though the uniform elongation was not observed in the tensile test. Considering the plastic instability of ultra-fine crystalline materials, it is clearly suggested that the reduction of area is a suitable indication of toughness than the area under the curve. In case of MA-8, surplus oxygen entrapped during MA, which does not form of ZrO₂, may be present in the form of oxide of Fe or Cr, or may be distributed at the grain boundaries as contamination, which may lead to a decrease in the toughness of the specimen.

Figure 14 shows the Charpy test specimens. In case of MA-1, 2, 3 and 4 with higher impact value, cracks advancing along the longitudinal direction are much observed for higher strength specimen. Kimura, *et al.* reported that the 1500 MPa grade ultra fine grained steels with fiber texture of $\langle 110 \rangle$ // Rolling Direction (RD) manufactured by severe



Fig. 14 Appearance of the specimens after Charpy impact test.



Fig. 15 Inverse pole figure in the cross-section of plane that are extruding direction.

plastic deformation and heat treatment shows high Charpy impact value and same fracture mode as those obtained in this study. It was suggested that the absorbed energy is as high as uE = 165 J in the transverse RD, since the fracture occurs as delamination of the fiber texture.²⁴⁾ The inversed pole figure in the transverse cross section of MA-1 is shown in Fig. 15. In this case, the fiber texture with $\langle 110 \rangle$ // Extruding Direction is also formed, which might be the reason for the high ductility.



Fig. 16 Cross section image of fracture surface by Charpy impact test.

On the other hand, impact value of MA-5, 6, 7 and 8 with higher oxygen content was very low with brittle fracture surface. Figure 16 shows the cross section around the fracture surface in MA-7. Primary powder boundary is clearly observed and crack grew along the boundaries. It is considered that the oxide layer on the primary powders or tiny voids at the boundaries may affect the toughness of powder metal products.

4. Conclusions

Ultra-fine crystalline ferritic stainless steels were made using MA method and hot extrusion. The effects of Zr oxide on metallurgical and mechanical properties were investigated. The results obtained are summarized as follows:

- (1) Zr containing Fe-12Cr, which is made via MA and hot extrusion shows an average grain size of $0.36 \,\mu$ m, whereas only 1 μ m is achieved via just extrusion. The dispersoid is identified to be Zr oxide or carbide, which is formed during hot extrusion with oxygen entrapped during MA and dispersed preferentially on the grain boundaries. Extrusion temperature and ZrO₂ content significantly affect the resultant grain size.
- (2) Zr containing Fe-12Cr follows the Hall-Petch relation up to a grain size of 100 nm and the gradient and strength value agree with those of pure iron, hence the grain refinement seems to be the dominant strengthening mechanism. The specimens with Zr additive show higher toughness of around 3 MJ/m² up to a tensile strength of 1500 MPa when compared to the commercial steels of JIS SUS410 and PH-stainless steel.

(3) Considering the plastic instability of ultra-fine crystalline materials, it is clearly suggested that the reduction of area is a more suitable indication of toughness than the area under the stress-strain curve. One of the issues is that the oxide layer on the primary powder or tiny voids at the boundaries may affect the toughness especially for higher oxygen containing steels.

Acknowledgement

This research was supported by the New Energy and Industrial Technology Development Organization (NEDO).

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