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Magnetic Properties of Austenitic Stainless Steel 316l and 316lvm after High Temperature Gas Nitriding Treatment

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Abstract

Biometallic materials for implant devices not only have to good corrosion resistance but also stable nonmagnetic properties. Various method have been developed for enhanced the corrosion resistance i.e low temperature gas nitriding treatments. Unfortunatelly, low temperature gas nitriding produce weakly ferromagnetic due the presence of expanded austenitic phases. Another treatments methods which is capable for improvement the mechanical properties is high temperature gas nitriding. However, the evaluation of magnetic properties of austenitic stainless steel 316L and 316LVM not yet investigation. The evaluation of magnetic properties of austenitis stainless steel 316L and 316LVM after high temperature gas nitriding treatments have been succesfully done. The magnetic properties are evaluated by vibrating sample magnetometre (VSM) test. The magnetic properties such as magnetic remenance, magnetic saturation and magnetic permeability are improved. As treated 316L and 316LVM have more stable non-magnetic properties and they more safe and compatible for MRI test.

Keywords: 316L, 316LVM, high temperature gas nitriding, magnetic properties, MRI

1. Introduction

Austenitic stainless steel 316L and 316LVM are ones of metallic biomaterial that widely used as implants and medical devices. The corrosion resistance can be used as the parameter of their biocompatibility [1]. Recently, increasing of corrosion resistance is not only concern for improvement of metallic biomaterial but also stable non-magnetic properties. The development magnetic resonance imaging (MRI) as clinical imaging tools needs metallic biomaterial that has stable non-magnetic properties. The principal issues for MRI safety and compatibility are magnetically induced displacements and torque, RF heating and image artifact [2]. Implanted and medical devices from austenitic stainless steel meet the MRI safety and compatibility in the up to 1.5T systems. However the developments of new 3.0T MR system cause their MR compatibility being decrease [3, 4].

Various surface treatments such as gas nitriding, ion implantation, and plasma nitriding have been successfully applied to enhance their corrosion resistance. These treatments usually have been conducted at low temperature or below the austenite temperature. Low temperature nitriding produces thin layer of expanded austenite phase which improve their hardness and corrosion resistant. This expanded austenite phase is ferromagnetic [5, 6]. Furthermore this phase cause weakly ferromagnetic properties of nitride austenitic stainless steel.

In the previous research, HTGN treatment increase the corrosion resistance of 316L and 316LVM [7, 8]. However the effect of HTGN treatments on the magnetic behavior of its materials not yet investigation. This papers deal with the effect of HTGN treatment on the magnetic properties of 316L and 316LVM.

2. Materials and methods

Specimens were prepared from 316L and 316LVM plate. The chemical compositions of the specimens are shown in table 1. Specimens were rinsed using ultrasonic cleaner in acetone as soaking medium prior HTGN treatments in order to remove oil and debris. HTGN treatments were carried out at modified three zone heating chamber of vertical furnace (Carbolite® type TZF 15/50/610). The furnace equipped with a precision digital pressure controller in order to maintain the pressure in the tube during treatments. Figure 1 shows the HTGN furnace that used during treatments.

Table 1. Chemical composition of specimens (%wt)

	C	Cr	Ni	Mo	Mn	Si	Fe
316L	0.01	15.5	11.8	1.24	1.23	0.47	balance
316LVM	0.01	17.3	15.5	1.73	1.67	0.42	balance

Specimens were inserted to the furnace tube, vacuumed to 10 Pa for 15 minute then flushed using nitrogen gas at 1000 ml/min for 15 minute prior heated. Nitrogen gas flowed continuously at 100 ml/min until treatment temperatures achieved. During process, the pressure inside the furnace tube maintained at 0,3atm. The temperature treatment was chosen at 1050 ^{o}C and holding time for 15 minutes. This temperature and holding time were selected as optimum process variables resulted from previous experiments [7, 8]. In the end of heating processes, the specimen was quenched in the water.

Magnetic properties were evaluated using vibrating sample magnetometer (VSM). As received and treated specimens processed in to powder using low speed saw before test. VSM test conducted by means exposure the powder in the magnetic field from -1 to 1 T. Magnetic moment (emu) recorded during VSM test. After HTGN treatments, treated specimens 316L and 316LVM were evaluated by XRD. The XRD test conducted at Shimadzu type 7000s. Scan range was chosen at 10 – 90^{o} and scan speed 2degree/min. XRD spectrum compared with crystallography open database (COD).

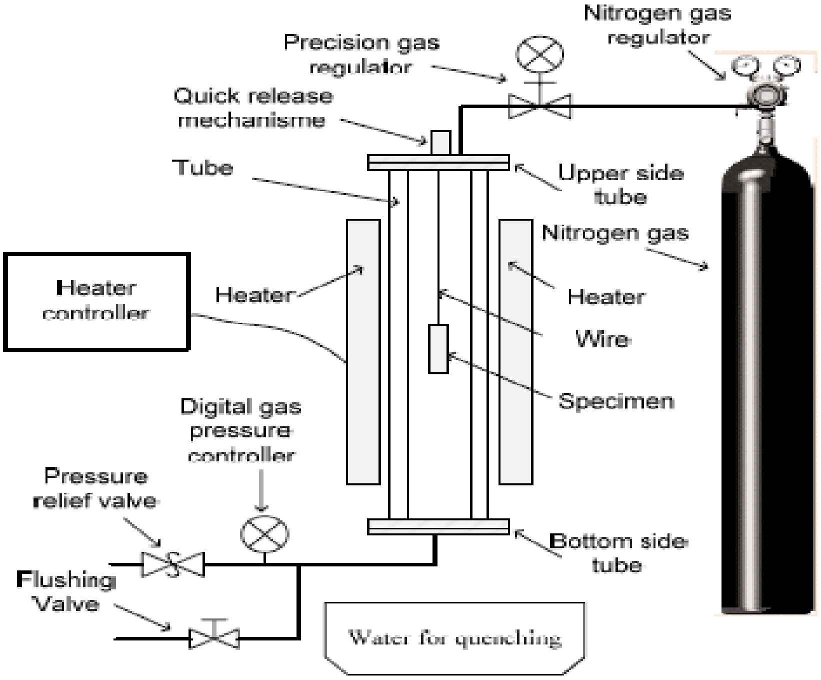


Figure 1. Modified three zone Carbolite® furnace for HTGN treatments

3. Results and analysis

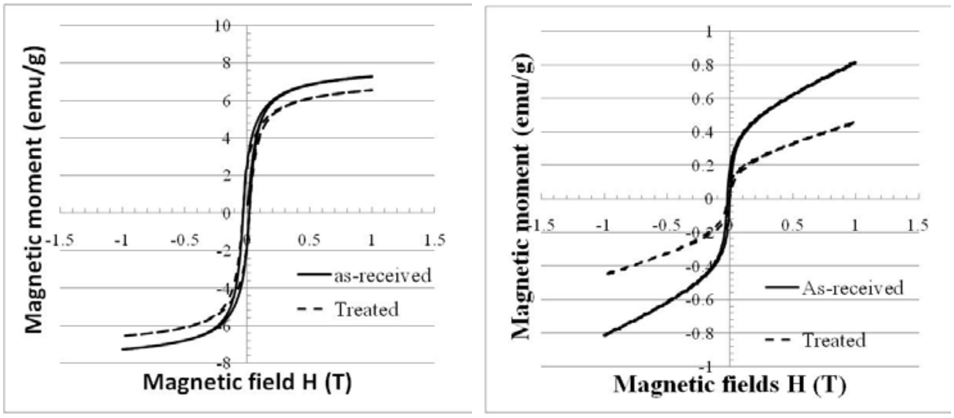
Figure 2 shows the magnetization curve from VSM test. As received 316LVM has magnetization curve is lower compared to the as-received 316L. It indicates that vacuums melting not only enhance the corrosion resistance but also improve the non-magnetic properties of 316LVM. Nonmagnetic properties of austenitic stainless steel increase with cleanliness and homogeneity.

Nonmagnetic properties of 316L and 316LVM are resulted from austenite phase. In the Fe-C alloys, austenite phase is only present at high temperature. The addition of austenite stabilizer elements such as Ni, Mn and N are caused the austenite phase stable until room temperature. However, this phase may transform into martensite phase if excessive cold working being applied. Martensite phase has strong ferromagnetic properties. The present of martensite phase at the austenitic stainless steel change the non-magnetic into weak magnetic properties. As received specimens are from plate which has been cold rolled during production. Martensite phase may present at the as-received specimens that posse weak magnetic properties. Heat treatments at the proper temperature can eliminate the martensite phase.

The present of the other phase in austenitic stainless steel such as (Fe, Cr, Mo)N compound has the same effect on the magnetism properties with martensite phase. Increasing nitrogen contents after HTGN treatment may produce that alloys. A proper selected variable process eliminate the present of these compounds [9].

Magnetization curve both treated specimens for 316L and 31LVM shows reduced the magnetic moments. It indicate that HTGN treatment not only increase the corrosion resistance but also improve the non-magnetic properties. However, the improving of nonmagnetic properties of 316LVM is higher than 316L. This phenomenon can be explained by dissolved magnetic phase during treatments and (Fe, Cr, Mo)N are not produced during treatments.

The comparison of magnetic properties as received and treated specimens are tabulated at Table 2. The magnitude of the magnetic saturation (M_S), magnetic remanence (M_R) and magnetic coercive ((H_C) decrease significantly. Magnetic susceptibility (κ) which is the slope of linier portion of hysteresis curve is also decreases. The magnetic permeability (μ_r) which is the parameter of non-magnetic stability is also decreases. Nitrogen is strong austenite stabilizer. The increasing nitrogen contents enhance the stability of non-magnetic properties of treated specimens.



a. 316L b. 316LVM

Figure 2. Magnetization curve of specimens

Tabel 2. The magnitude of M_s, M_r, H_c, κ and μ_r

	316L		316LVM	
	As-received	T = 1050^{o}C & t = 15 minutes	As-received	T = 1050^{o}C & t = 15 minutes
M_s	7,29 emu/gr	6,54 emu/gr	0,78 emu/gr	0,431 emu/gr
M_r	2,52 emu/gr	2,33 emu/gr	0,16 emu/gr	0,079 emu/gr
H_c	2,11 x 10^6 Oe	1,41 10^6 Oe	9,92 x10^{-2} Oe	8,17 x10^{-2} Oe
κ	0,0042	0,0039	0,0006	0,0002
μ_r	1,0042	1,0039	1,0006	1,0002

The phase of 316L and 316LVM after treatments are still austenite as shows at Figure 3. Increasing nitrogen contents is limited to the solubility of austenite phase. The temperature of HTGN treatment is at the austenite temperature. The only phase at treatment temperature is only austenite, the other phase such as martensite, ferrite and pearlite that may presents at room temperature dissolve during heating. Quenching process at the end of treatments produces austenite phase into room temperature.

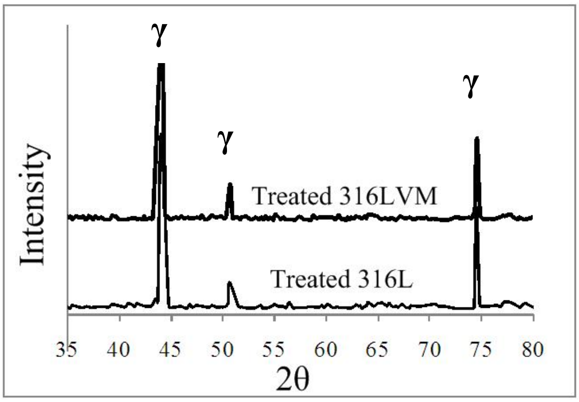


Figure 3. XRD spectrum

The HTGN treatments does not produce the formation of (Fe, Cr, Mo)N compounds which may presents due to the increasing the nitrogen contents.. After solubility limit is achieved, nitrogen cannot continue to diffuse to the stainless steel. Diffusion process starts at the surface of specimens. The nitrogen contents at the surface may reach the limited solubility immediately. Longer holding time will produce diffusion more depth. So, the short holding time is enough to increase the nitrogen contents at the surface. It results higher corrosion resistance than as received specimens [8]. The solubility limit of nitrogen cause the formation of (Fe, Cr, Mo)N cannot obtained by HTGN treatments for austenitic stainless steel. Figure 3 shows that the only austenitic phase presents at the specimens after HTGN treatments.

4. Conclusion

HTGN treatments for austenitic stainless steel 316L and 316LVM at treatments temperature 1050 ^{o}C for 15 minutes enhance the stability of their non-magnetic properties. HTGN treatments can dissolve the magnetic phase and does not produces (Fe, Cr, Mo)N compounds. The enhancing of nonmagnetic properties indicates that treated specimens are more biocompatible and MR safe than as-received samples.

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