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Evaluation on the Effect of HTGN Treatments on theCorrosion Resistance and Magnetic Properties of Austenitic Stainless Steel 316L and 316LVM

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Abstract

High temperature gas nitriding (HTGN) is the new methods to enhance the properties of stainless steel. The HTGN process is able to diffuse the nitrogen atom into stainless steel. Increasing the nitrogen concentration produces higher corrosion resistance. Stainless steel for implant and medical devices such as 316L and 316LVM not only have to high corrosion resistance but also have to magnetic properties stabilities. Evaluation of corrosion and magnetic properties for austenitic stainless steel 316L and 316LVM after HTGN treatments was successfully done. The corrosion resistance not only significantly increases but the stability of austenite phase is also increases. Therefore HTGN treatments is suitable for improving the corrosion resistance for 316L and 316LVM which used as implant material.

Keywords: austenitic stainless steel 316L and 316LVM; HTGN treatments; implant material

1. INTRODUCTION

Metallic biomaterials like austenitic stainless steel 316L and 316LVM are frequently employed as implants and medical equipment. They exhibit strong mechanical qualities and high corrosion resistance. Metallic biomaterials' resistance to corrosion can be utilised as a measure of their biocompatibility. It is more biocompatible to have high corrosion resistance. Nevertheless, the corrosion resistance of 316L and 316LVM decreases in corrosive body fluid environments, low oxygen levels, and heavy loads like bone fixation. Products that produce corrosion have any negative consequences on the body [1].

To increase their corrosion resistance, several surface treatments such gas nitriding, ion implantation, and plasma nitriding have been successfully used. The majority of the time, these treatments have been carried out at or below the austenite temperature. Its hardness and corrosion resistance are improved by the thin layer of enlarged austenite phase produced by low temperature nitriding. Ferromagnetism characterises this extended austenite phase [2, 3]. Moreover, this phase gives rise to the nitride austenitic stainless steel's weakly ferromagnetic characteristics.

~~Lately, improving corrosion resistance has become important for both stable~~ non-magnetic characteristics and the enhancement of metallic biomaterials. A stable non-magnetic metallic biomaterial is required for the development of magnetic resonance imaging (MRI) as a clinical imaging technique. Magnetically generated displacements and torque, radio frequency (RF) heating, and picture artefact are the main concerns for MR safety and compatibility [4]. Austenitic stainless steel implants and medical equipment are safe and compatible with MRI up to 1.5T systems. However, the advancements of the current 3.0T MR system have reduced their MR compatibility [5, 6].

phase or another ferromagnetic phase such as (Fe, Cr, Mo)N is high temperature gas nitriding (HTGN) [7]. HTGN treatment is thermo chemical process which able to diffuse the nitrogen atom into stainless steel. Hardness and corrosion resistance significantly improve by this treatment.

The early work for elimination the magnetic properties using HTGN treatment has developed. Weakly magnetic properties of free nickel high nitrogen austenitic stainless steel due the present of delta ferrite can be eliminated by HTGN treatments [8]. Although HTGN treatment for stainless steel has been investigate by many researchers [7, 9, 10] but the effect of HTGN treatments on the magnetic behavior of 316L and 316LVM not yet investigation. This papers deal with the effect of HTGN treatment on the corrosion and magnetic properties of 316L and 316LVM.

2. METHODOLOGY

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.

Table 2.1 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 [11, 12]. After heating process, specimen was quenched in the water.

As received and treated specimens were cut and machined to produce 1.4 mm disc. The discs were gently polished in order to remove the scale using metal polish. These discs produced 1 cm^2 area in the corrosion test. The discs were rinsed by acetone prior corrosion test. The corrosion test was carried out at the surface of disc using polarizations resistance corrosion techniques in ringer solution as corrosion medium at temperature 37^{o}C. For the first running test, the initial potential test and final was selected -20mV vs Ecorr and +20mV vs SCE respectively and the potential scan rate was 0.1mV. Each specimen tested for three times to determined Icorr.

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).

3. RESULT and DISCUSSION

Figure 3.1 shows the polarization curve obtained from corrosion test. Polarization curve for treated specimen both 316L and 316LVM are shift to up and left. It indicates that treated specimen has more corrosion resistance than as-received ones. Corrosion resistance of as received 316LVM is higher than as-received 316L. However, the corrosion resistance for treated specimen of 316L is close to the as received 316LVM. It indicates that HTGN treatment is able to enhance the corrosion resistance of 316L.

Table 3.1 show the Ecorr and icorr for as-received and treated specimens. Corrosion resistance of as received 316L increases from 0.0228mpy up to 0.0011mpy after HTGN treatment. Corrosion resistance of as received 316LVM increases from 0.0013 up to 0.0002mpy after treatments. The increasing of corrosion resistance produced by HTGN treatments of 316L is higher than for 316LVM. HTGN treatment for 316L is more effective to increasing the corrosion resistance than 316LVM.

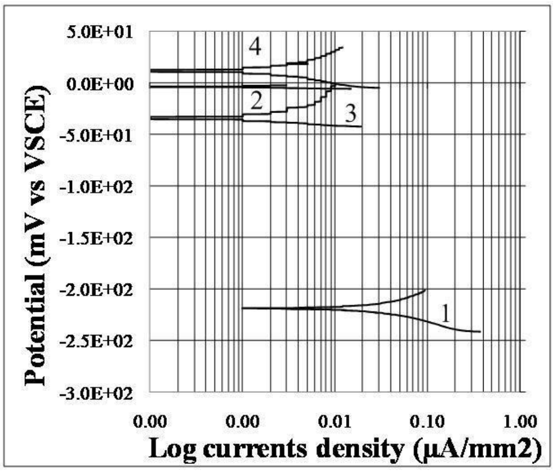


Figure 3.1 Polarization curve of as-received and treated specimens
1: as-received 316L, 2: treated 316L, 3: as-received 316LVM & 4: treated

Specimens		E_{corr} (mV)	i_{corr} (µA/cm^2)	Corrosion rate (mpy)
316L	As-received	-218.46	0.06	0.0228
	Treated	-119.48	0.00040	0.0011
316LVM	As-received	-33.43	0.00041	0.0013
	Treated	-39.11	0.00006	0.0002

The corrosion resistance of stainless steels may predicted by pitting resistance equivalent number (PREN). The magnitude of PREN only depends on the chemical composition of stainless steel mainly for Cr, Mo and N. The corrosion resistance increase with PREN. The formulae of PREN is %Cr + 3,3x%Mo + (20 – 30)%N. However, the corrosion resistance of stainless steel actually not only depends on the chemical composition but also the cleanliness and homogeneity of their micro structure. Corrosion resistance increases with cleanliness and homogeneity.

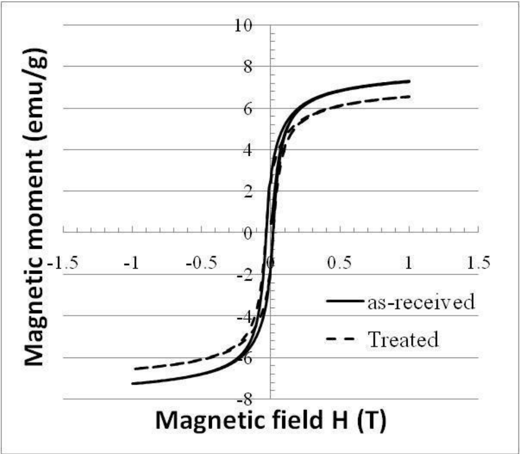
The chemical composition of 316L and 316LVM is not significantly different. There for the magnitude of PREN is also identically. Although the magnitude of PREN for 316L and 316LVM is not significantly different, the corrosion resistance of as received 316LVM is higher than as received 316L. Type 316LVM is produced by remelting 316L in the vacuum environment. The vacuum casting process produces micro structures which increase cleanliness and homogeneity [13, 14]. Segregation and impurity are significantly reduced and lead to higher corrosion resistance.

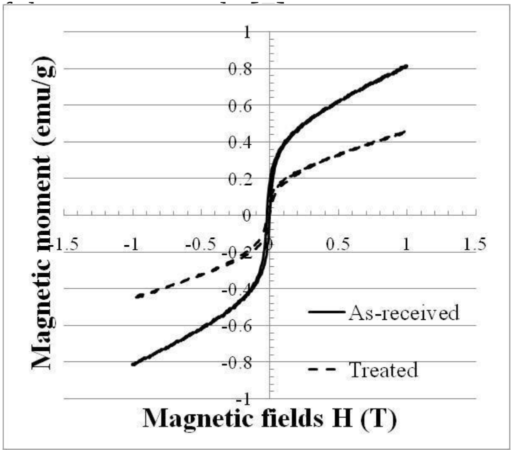
Treated 316LVM has corrosion resistance close to the as-received 316LVM as indicate that polarization curve is close each other. Corrosion process do at the surface, there for the increasing corrosion resistance depends on the chemical composition at the surface. HTGN treatments increase the nitrogen contents. The diffusion process starts at the surface and become take place at the depth region. The magnitude of PREN at the surface increase with nitrogen contents at the surface. Increasing nitrogen contents at the surface cause the increasing corrosion resistance of the specimens.

Figure 3.2 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. Non-magnetic properties of austenitic stainless steel increase with cleanliness and homogeneity.

Non-magnetic 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 selecte





a. 316L b. 316LVM

Figure 3.2 Magnetization curve of specimens

Magnetization curve both treated specimens for 316L and 31LVM shows reducedthe magnetic moments. It indicate that HTGN treatment not only increase the corrosion resistance but also improve the non-magnetic properties. However, the improving of non-magnetic 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 temperature of HTGN treatment is at the austenite temperature. In this temperature the only phase is austenite, the other phase such as martensite phase that may presents at room temperature dissolve during heating. Quenching process at the end of treatments produces austenite phase until room temperature.

The treatments does not produce the formation of (Fe, Cr, Mo)N compounds as indicate from XRD spectrum at Fig. 3 which may presents due to the increasing the nitrogen contents. Increasing nitrogen contents is limited to the solubility of austenite phase. 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.

The solubility limit of nitrogen cause the formation of (Fe, Cr, Mo)N cannot be obtained by HTGN treatments for austenitic stainless steel.

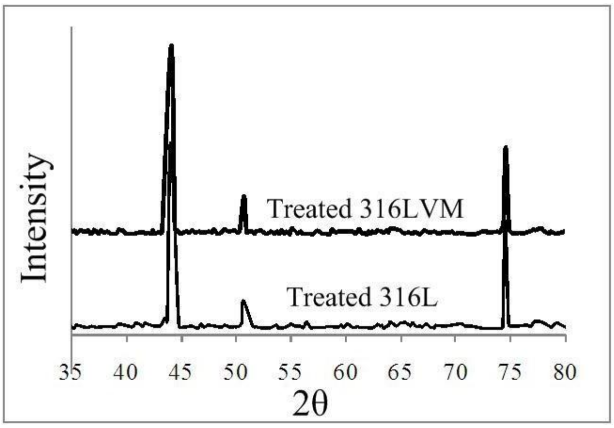


Figure 3.3 XRD spectrum specimens after HTGN treatments

4. CONCLUSION

Both corrosion resistance and non-magnetic characteristics were improved by HTGN treatments for austenitic stainless steel 316L and 316LVM at treatment temperature 1050 oC for 15 minutes. Because HTGN can dissolve the magnetic phase and doesn't form (Fe, Cr, or Mo)N compounds, the non-magnetic properties have improved after treatments. Corrosion resistance and non-magnetic characteristics being improved suggest that treated specimens are more MR safe and biocompatible than untreated samples.

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