



Research Article

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## Corrosion behavior of Austenitic stainless steel 316L and 316LVM after high temperature gas nitriding

Soekrisno Raden, Suyitno Dharmastiti Rini and Suprihanto Agus

*Mechanical and Industrial Engineering Department, Gadjah Mada University, Jl. Grafika #2, Bulaksumur, Yogyakarta, Indonesia*

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### ABSTRACT

*Austenitic stainless steel 316L and 316LVM are widely used as implant materials due to the good mechanical properties and corrosion resistance. However at highly stressed and oxygen-depleted environment, it still susceptible to corrosion. Various methods have been developed to enhance the corrosion resistance of stainless steel such as high temperature gas nitriding. High temperature gas nitriding for 316L and 316LVM has been successfully done. This process is conducted at temperature 1050, 1100 and 1200°C for 15 and 30minutes before quenching in the water. Polarization corrosion test showed that the corrosion resistance was significant improved for treatment at temperature 1050°C and holding time 15minutes.*

**Keywords:** corrosion resistance, 316L, 316LVM, high temperature gas nitriding

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### INTRODUCTION

The first stainless steel utilized for implant fabrication was 18-8. Later 18-8sMo stainless steel was introduced which contains a small percentage of molybdenum to improve the corrosion resistance in chloride solution[1]. This alloy became known as type 316 which has carbon contents 0.08% (wt). In order to minimize the sensitization and better corrosion resistance, the carbon content is reduce to maximum 0.03% (wt). This alloy is known as austenitic stainless steel 316L. Austenitic stainless steel 316L is non-magnetic. It cannot be hardened by heat treatments but can be hardened by cold working. A wide range of mechanical properties exists such as softer for annealing conditions or greater strength and hardness for cold working conditions. Selecting of these materials for implant fabrication must be careful because it still susceptible to corrosion under highly stressed and oxygen-depleted environments such as the contacts under the screw of the bone fracture plate[2].

The corrosion process of implants can be done by fretting, crevice and pitting corrosion. The product of corrosion is detrimental to the body which can lead to irritation, allergy and infection[3]. Various methods were developed to enhance the corrosion resistance of 316L such as surface treatments. The well known surface treatments which commonly applies for example surface passivation [4,5], sandblasting [6,7], plasma nitriding [8], carbonitriding, etc. The other methods is improved the cleanliness and homogeneity [9]. Special casting process includingpressurized electro slag remelting (PSER) in the vacuum conditions in order to enhancethe cleanliness and homogeneity. Austenitic stainless steel 316L which fabricated by these special casting process is became known as 316LVM.

Corrosion resistance of 316LVM is significantly enhanced compared to its predecessor 316L due to the excellent micro cleanliness and structural homogeneity [9,10]. This stainless steel is suitable for the production of both temporary and permanent implants. However, austenitic stainless steel 316LVM is more susceptible to corrosion under body fluid compared to the titanium and titanium alloy [2].

Improvement of the corrosion resistance of stainless steel can be performed by thermo chemical treatment such as nitriding. Nitriding is a process which adds nitrogen atom in the surface of specimens by diffusion. Low temperature nitriding improve both its corrosion resistance and hardness due the formation of thin layer about few micrometer of S-phase[11]. Special surface preparation such as surface activation is needed before nitriding process. Surface activation is used to remove the surface passivity film which act as obstacle diffusion process. Another prospective nitriding process which does not need special surface treatments is high temperature gas nitriding (HTGN)[12].

The principle of HTGN treatments is holding the stainless steel at temperature 1050-1200°C for certain time in the nitrogen gas atmosphere prior quenching[13]. These treatments produce thicker diffusion depth than low temperature nitriding. Depending on the composition of stainless steel, temperature treatments, holding time and nitrogen gas partial pressure, microstructure and depth of diffusion can be produced. Ferritic stainless steel can be modified in to martensitic or austenitic by HTGN treatments[14]. The corrosion resistance is improved due the increasing of nitrogen contents[15].

Although HTGN treatment for stainless steel has been investigated by many researchers, the comparison of the effect of HTGN treatments on the corrosion behavior for 316L and 316LVM not yet investigated. This paper discuss the corrosion behavior for austenitic stainless steel 316L and 316LVM after short duration of HTGN treatments.

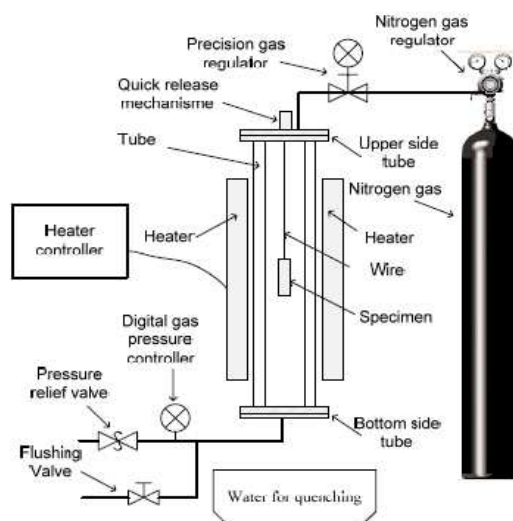
## 2. Experimental Procedures

Specimens were prepared from 2 mm thick plate of 316L and 4 mm thick plate of austenitic stainless steel 316LVM. 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 was carried out at modified three zone heating chamber of vertical furnace (Carbolite® type TZF 15/50/610) which equipped with a precision digital pressure controller as shown schematically at Figure 1.

**Table 1. Chemical composition (%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. Flushing valve opened and tube 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 and flushing valve then closed. The temperature treatment was chosen at 1050, 1100 and 1200°C. The nitrogen gas pressure maintained at 0.3 atm. using precision digital gas pressure controller equipment. If the gas pressure increased above 0.3 atm., the controller opened the solenoid valve automatically and reduced the gas pressure back to 0.3 atm. The holding time was chosen for 15 and 30 minutes for 316L and 15 minute for 316LVM. This holding time were selected as optimum holding time which resulted from the previous experiments. After heating process, specimens was quenched in the water by opened the bottom side of tube and quick release mechanism.



**Fig. 1. Modified three zone Carbolite® furnace for HTGN treatments**

As received and treated specimens were cut and machined to produce 14 mm disc. The discs were gently polished in order to remove the scale using metal polish. These discs produced 1 cm<sup>2</sup> 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°C. For the first running test, the initial potential test and final was selected -20mV vs E<sub>corr</sub> and +20mV vs SCE respectively, and the potential scan rate was 0.1 mV. Each specimen tested for three times to determine the I<sub>corr</sub>.

## RESULTS AND DISCUSSION

The effect of treatments temperature of HTGN process for 15 minutes holding time on the corrosion of 316L is shown in Fig.2. The curve is shifted to the left and up compared to the as-received sample. E<sub>corr</sub> for treated sample decreases after treatments. It indicates that the treated sample is better corrosion resistance compared to the as-received. The corrosion resistance increases due to the diffusion of nitrogen in to the sample.

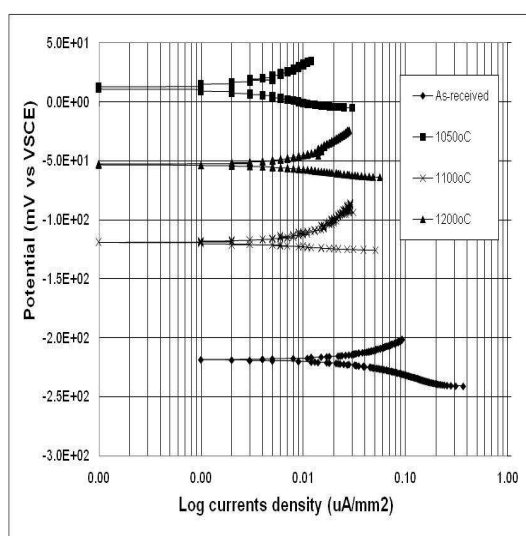


Fig. 2. Polarization resistance curve for 15 minutes holding time

Fig. 3 shows the effect of 30 minutes holding time on the corrosion resistance for 316L. Treated sample at 1050°C has higher corrosion resistance compared to the others. This result is identical to the 15 minute holding time. The highest corrosion resistance is achieved at treatments temperature at 1050 followed by 1200 and 1100°C. The corrosion curve for treatments temperature is close to the corrosion curve of treatment temperature at 1050°C. It indicates that for 30 minutes holding time, the corrosion resistance for treated sample at 1050°C is identical to 1200°C.

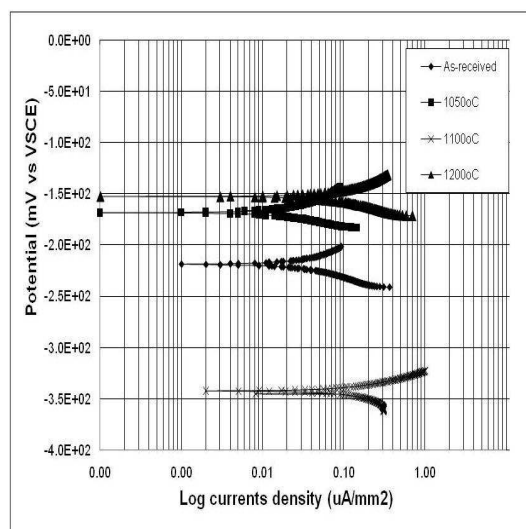
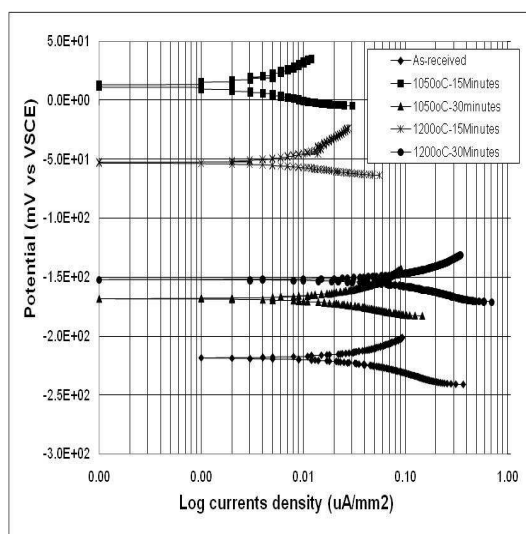


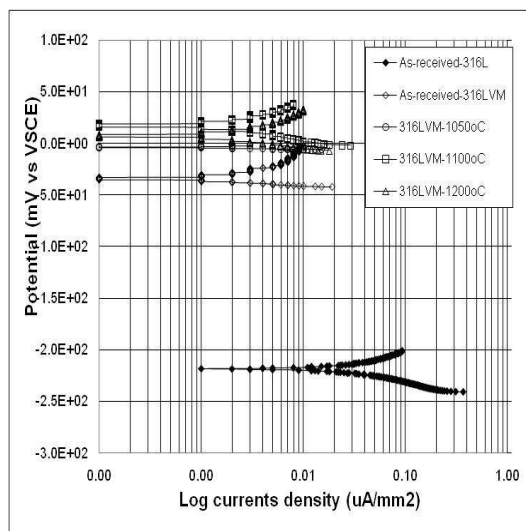
Fig. 3. Polarization resistance curve for 30 minutes holding time

Fig. 4 shows the corrosion curve for 15 and 30 minutes holding time and treatment temperature at 1050 and 1200°C. The corrosion resistance for 30 minute holding time is less than 15 minutes holding time for all treatments temperature. The short holding time and lower treatment temperature produce the highest corrosion resistance. This fact showed that the best corrosion improvement for 316L is achieved at 15 minutes holding time and treatment temperature at 1050°C.



**Fig. 4. Polarization resistance curve for 15 and 30 minutes holding time and treatment temperature 1050and 1200°C**

Fig. 5 shows the results of corrosion test for 316LVM. The  $E_{corr}$  for 316LVM is less than 316L. It indicates that the corrosion resistance 316LVM is higher than 316L. The corrosion resistance of 316LVM after HTGN treatment is higher than as-received, but the increasing of corrosion resistance does not higher than 316L. The chemical composition of austenitic stainless steel 316LVM is identical to the 316L, but the cleanliness and homogeneity is improved. It produces higher corrosion resistance than 316L.



**Fig. 5. Polarization resistance curve for 15 minutes holding time for 316LVM**

The effect of nitrogen on the corrosion resistance of stainless steel can be predicted using pitting resistance equivalent number (PREN). The formula of PREN is  $\%Cr + 3.3\%Mo + (10 - 30)\%N$ . Increasing PREN indicates that stainless steel is more corrosion resistance. PREN formula shows that nitrogen is more effective to improve the corrosion resistance of stainless steel.

HTGN treatment is thermo chemical process which adds nitrogen atoms by diffusion process. The amount of nitrogen atoms diffused in to sample depends on the chemical composition of stainless steel, nitrogen partial gas pressure, treatments temperature and holding time[12,14].The higher treatments temperature and longer holding time results more nitrogen atoms diffused in to sample, raises the magnitude of PREN and increases the corrosion

resistance. Despite increasing treatments temperature results more nitrogen atom diffused in to sample, the corrosion resistance of 316L does not increase too. It shows from Fig. 2, the corrosion resistance of treated sample at 1050 is higher than at 1100°C. However, the corrosion resistance of 316LVM is identical for all treatments temperature.

Corrosion is the process releasing metal ion which occurs at the surface. The corrosion resistance of the sample depends on the surface passive film and its stability. Corrosion occurs if the surface passive film is broken. Strong and stable surface film produce higher corrosion resistance of materials. Because corrosion depends on the conditions at the surface, the effect of diffusion depth does not significant. Longer holding time and higher treatments temperature in HTGN only produces more diffusion depth.

The nitrogen atom which diffused in to sample is from dissociation process of nitrogen gas molecules at the surface due to the present of Fe which acts as catalyst. Nitrogen atoms then diffuse at the surface. It produce that the concentration of nitrogen atoms at the surface for short holding time is higher than at the region below the surface. It can be assumed that the diffusion rate of nitrogen in the sample is slower than at the surface. Furthermore, the concentrations of nitrogen at the surface reach its solubility limits. Increasing holding time and treatments temperature do not increase the nitrogen contents at the surface, but it increase the concentration of nitrogen in the region below the surface.

The solubility of nitrogen in the stainless steel decreases with temperature[14]. The higher solubility is obtained at 1050 than 1100 and 1200°C. Thus, the highest magnitude of PREN and the corrosion resistance are achieved at treatments temperature 1050°C. The effects of solubility limit at the sample explain why the highest corrosion resistance is obtained at 15 minute holding time and treatments temperature at 1050°C.

## CONCLUSION

HTGN treatment enhances the corrosion rate of austenitic stainless steel 316L and 316LVM. The corrosion curve after HTGN treatments is shifted to the left and up which indicates that corrosion resistance increases after treatments. The highest corrosion resistance is obtained at 15 minutes holding time and treatment temperature at 1050°C.

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