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Effects on the Long-Term Aging of Low Temperature Salt Bath Nitrided AISI 321 Stainless Steel

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Abstract. AISI 321 stainless steel was salt bath nitrided at 430K for 8 hours, then being long-term aging treatment at different temperature. To study the changes of microstructures and performances of these samples by using X-ray diffraction (XRD), scanning electron microscopy (SEM), micro hardness tester, electrochemical workstation, and universal tensile testing machine. Experimental results showed that a nitrided layer was formed on the surface with the thickness ranging from 7.6 to 22.5 μm varying with changing aging treatment time and temperature after dealing with salt bath nitriding. The thickness of the nitrided layer increased with increasing aging time and temperature. According to the fick's second law, the growth of the nitride layer is mainly due to the diffusion of nitrogen, as expected. The active energy of nitrogen atoms in AISI 321 stainless steel is 340.2kJ/mol by calculated form Thermodynamics formula. The surface micro hardness significantly increased to nearly 1430 Hv0.098, which is about 4 times harder the non-treated material, and then gradually reduced. The corrosion resistance of the 321 stainless steel is increases with proper aging treatment, but when the aging time more than 15.552×10^{6}s, the corrosion resistance decrease. The tensile properties of AISI 321 ASS of strength decrease but the plastic increase a little after salt bathing nitriding and aging.

1. Introduction

Austenitic stainless steel AISI 321, because of their various good performances such as excellent corrosion resistance [1-3], radiation sensitivity and mechanical processing used in the production of control rod components, water pile frameworks and fuel components screw components. However, these components run in fluid medium environment in pile system, mutual contact and fluid flow will lead to the failure of abrasion of the components. The low hardness and wear resistance restrict its use as key components in the reactor and harm to the safety of relevant components. The data shows that there have been two accidents which two control rod mill is broken and fell leaded to a serious threat to reactor safety in the end of last century. Framatome found that low temperature strengthening is the best choice to improve corrosion and wear resistance of austenitic stainless steels AISI 321 in the reactor after dealing with low temperature strengthening, the hardness of stainless steel increased significantly without degrading their corrosion resistance. Therefore enlarge the using range of the stainless steel [4-6].

Due to expanded the austenite (S phase) in the nitriding layer is not stable on the thermodynamic, the microstructure would changed within long-term serving under the temperature of reactor. Li and Dong [7-10] did research about the influence of aging to the corrosion resistance of austenitic. The study shows that once interstitial precipitated from the S phase, the corrosion resistance of austenitic would be decreased. Yan's literature [16] indicated that expanded austenite of AISI 2205 duplex stainless steel was decomposed and ε-nitride precipitated subsequently when the nitriding time prolonged up to 16 hours. Previous report found that when a large number of interstitial atoms make the lattice expansion to a certain amount, which greatly reduces the volume of mismatch precipitated; and alloy elements diffuse through the crystal defects, is conducive to the precipitation of interstitial compounds [12] .And alloying elements diffused through the crystal defects, which is conducive to the precipitation of interstitial compounds.

Unfortunately, for the expanded austenite in the process of long-term aging temperature and influence on the performance of decomposition is not clear. This will limit the further application of austenitic steel. Therefore, we simulated the operating temperature of reactor, and analyzed systematically the microstructure and performance of expanded austenite after a long period of aging time. Further research on this project will not only help improving the nuclear safety of stainless steel components, but also benefit the development of low temperature surface engineering technology of stainless steel.

2. Experimental method

The chemical composition of stainless steel AISI 321 in this research is shown in Table 1. The samples for the experiment were wire-cutting processing into dimensions of 20mm×10mm×2mm. Before liquid salt bath nitriding, the flat surface of each sample was ground by sand paper and ultrasonically cleaned with acetone, alcohol and distilled water in succession. Then the samples of stainless steel AISI 321 were dipped in the molten salt bath at 430K for 8h. After nitrocarburizing, these samples were put into heat treatment furnace respectively at 325K、350K、375K for 0 days, 14 days (1.2096×10^6s), 30 days (2.592×10^6s), 90 days (7.776×10^6s), 180 days (15.552×10^6s), 240 days (20.736×10^6s). After the aging treatment, samples were cleaned for 15min in the alcohol bath ultrasonic wave.

The salt used for salt nitriding experiment stainless steel AISI 321 samples was mainly made up of M_2CO_3 (M denotes elements of K, Na, Li), $CO(NH_2)_2$ and some trace components. CNO^- concentration in the salt was above 40%. The nascent nitrogen used in nitriding reaction from the dissociation of CNO^- : $4CNO^- \rightarrow CO_3^{2-} + 2CN^- + CO + 2[N]$. What's more, few nascent carbon which comes from the dissociation of CO : $2CO \rightarrow CO_2 + [C]$, also permeates into austenite structure together with nitrogen.

The cross-sections of nitrocarburized layers etched by reagent (HCl : HNO3 : H2O=2 : 1 : 1) were observed by optical microscope and the Type OLYMPUS BX61 scanning electron microscopic observation and Hitachi S4800 scanning electron microscopy (SEM) with the Oxford EDS tester. X-ray diffractometer type Dmax-1400 with CuK alpha radiation and a nickel filter were used to determine the phases present in the modified layer. A Vickers microhardness tester was used to measure the surface hardness under 0.098N loading for 15s.

Dynamic polarization experiments used commercial electrochemical system (Model CS310, Wuhan CorrTest Instrument Co. Ltd, China). The scan rate was 5 mV/s and the experiments were conducted in the 3.5 mass% NaCl solutions at room temperature (20K). The reference electrode was saturated calomel electrode (SCE), the counter electrode was platinum plate (Pt), and the samples were connected with the working electrode. The electrodes were prepared by epoxy cold resin mounting of specimens, leaving areas for exposure to the electrolyte of about 1-2mm.

Table 1. Composition of type austenite stainless steel AISI 321 (mass %).

Elements	C	Si	Mn	Cr	Ni	Ti	Fe
Mass.%	0.04	0.54	1.33	17.55	9.00	0.48	Bal.

3. Results and discussion

3.1. Metallography analysis

Figure1 shows the relationship between the change of the nitrided layer and the various aging time after salt-bath nitrided conditions. It is clearly shows a bright white layer on the metallographic section. The formation of white layer is due to nitrogen after salt bath nitriding processing. But the substrate does not. During aging treatment, In the middle of the white layer appeared a black thin line. It clearly presents that the depth of the nitrided layer increases with the increasing the aging time.Figure1c shows the depth of the total modified layer is increased to 15 μm after aging treatment for 7.776×10^6s at 375°C. During aging treatment, In the middle of the white layer appears a black thin line. After the stainless steel specimen is treated for 15.552×10^6s, some of the secondary precipitates transform along the grains boundaries. The corrosion resistances of precipitate zone become worse. (Figure 1d) It clearly shows that the nitrided layer and substrate is blurred owing to nitrogen atoms spread to the matrix of internal. As the aging time increases to 20.736×10^6s, the microstructure of the nitrided layer changes. Many cracks are observed in the surface of the nitrided layer because of the induced stress fields.

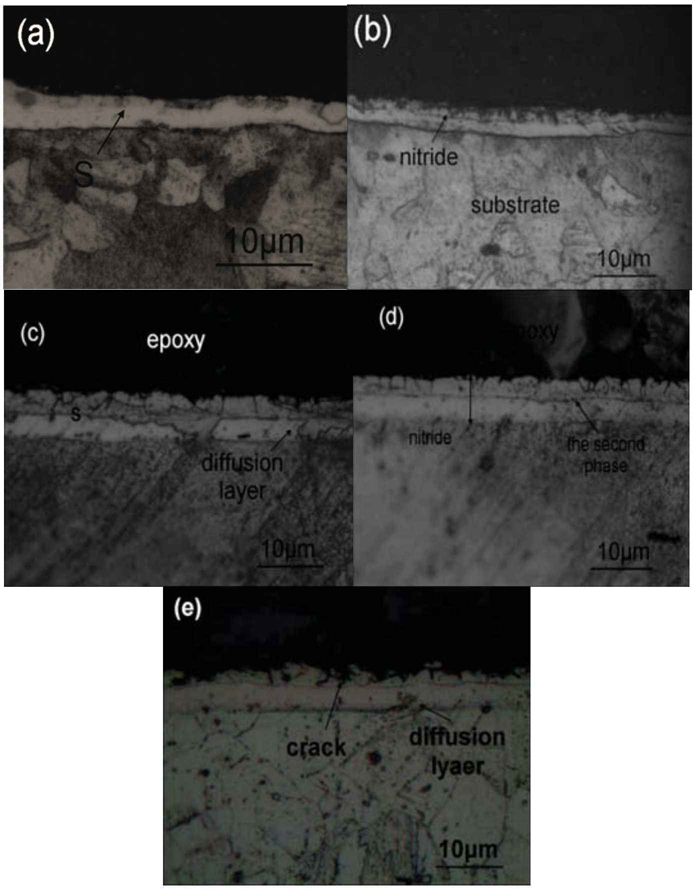

Figure 1. Optical metallography of low temperature salt bath nitrided AISI 321 ASS under various time aging in 375K (a: 0; b: 2.592×106s; c: 7.776×106s; d: 15.552×106s; e: 20.736×106s).

3.2. Diffusion analysis of the nitrided layer in in Stainless Steel AISI 321

The X-ray diffraction patterns at the nitrocarburized samples fewer than 350K aging with different times are shown in Figure 2. Under 350K aging environment, it can be seen from the figure that this sample shows austenite peak clearly. The peak skews obviously with the increase of the aging time. The nitrogen potential on the surface of the substrate is very high because of nitriding. N atoms' atomic radius of 0.074nm, approximately half of Fe atoms', so nitrogen atoms is spread more easily in Fe base lattice clearance. In a short period of time, nitrogen atoms still dissolve into the austenitic lattice. We can find that the peaks of γ (111), γ (200) and γ (220) shift toward the lower angles lightly. This research results obviously from the gradual distortion of the lattice's cubic symmetry [13]. The fcc grain lattice is supersaturated form by nitrogen when the nitrogen diffuses inward to the substrate. It is the process of austenite transformed into expended austenite, also called S phase. However, when the aging time more than 2.592×10^6s, nitrogen atoms is spread constantly to the matrix of internal and the concentration of nitrogen atoms is decrease gradually. The diffusion of nitrogen atoms is reaction diffusion and phase transformation diffusion. Nitrogen atoms constantly overflowed which dissolved in Fe base lattice clearance. When aging time up to 20.736×10^6s, gradual peak shift can be observed, and especially the (111) reflection is moved considerably toward higher angles. As the nitriding time extended, the lattice parameter become larger which consistent with the results from the other papers. Figure 3 shows the estimated lattice parameters and interlinear spacing in the peaks of α (1 1 1)from Bragg's law as a function of aging time. The lattice parameters of α (1 1 1) is 3.6919 in 1.2096×10^6s aging time. However, the lattice parameters reduce to 3.6302 under 20.736×10^6s aging time. This phenomenon of the lattice parameter results obviously is due to the gradual diffusion of the nitrogen atoms in the lattice with the different aging time and temperature. It can be seen that the thickness of the nitrided layer soars up with prolonged treatment time. The nitrided layer contains lots of supersaturated nitrogen atoms after salt bath nitriding. So the concentration of nitrogen atoms in the nitride layer increase. In the process of aging, the nitrogen atoms spread to the matrix of internal. Diffusion layer transforms into the nitrided layer with the increase of aging time, the thickness of the nitride layer increased constantly too. It shows a parabolic rate law expressed as the following equation: $D2 \propto Ct$, where D is the thickness of nitriding layer, C is a constant and t is treated time.

As is known to all, a parabolic relationship with d and t, d and t1/2 into a linear relationship, the slope uses it on behalf of diffusion velocity. It can be seen from Figure 4 that the nitrided layer was basically in a straight line. This result suggests that this diffusion process is compatible with Fick's second law [16]. With the increase of aging temperature, linear slope increase, that is to say, the diffusion velocity increase. The conclusion is consistent with the references which pointed out that the increase of temperature can improve the diffusion coefficient of nitrogen and get a thicker nitrided case.

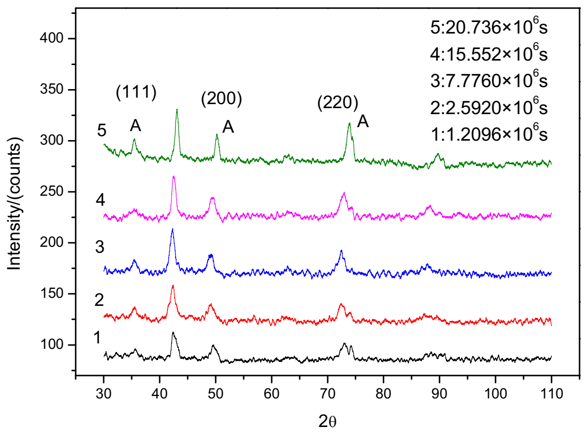

Figure 2. XRD Profile of different aging time of the nitrided AISI 321 Stainless Steel at 350°C.

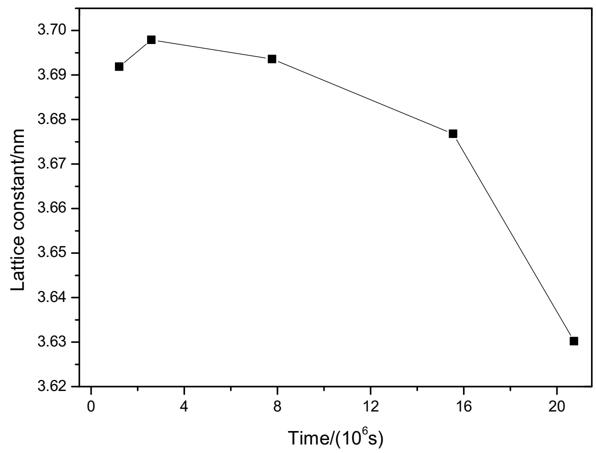

Figure 3. Lattice constant of S phase from (111) at different aging times.

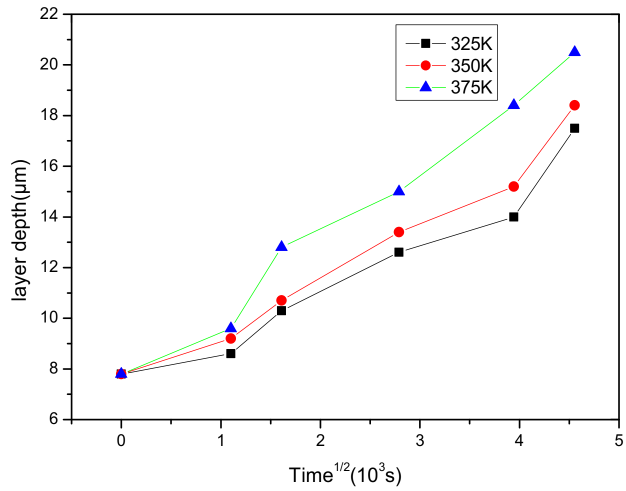

Figure 4. Change of nitrided layer thickness with aging time in AISI 321 ASS.

The microstructure morphology analysis suggests that it can forms different thickness of the nitrided layer by treated in different aging temperature. The nitrided layer thickness is 7.6μm after salt bath nitriding treatment at 430K for 8 hour. The thickness of the nitrided layer was increased with the aging time prolonging, the nitrided layer thickness is more than 20μm when the aging time after eight months. In the process of aging, the spread of the main ways for nitrogen atoms is reaction diffusion and phase transformation diffusion.

Diffusion of nitrogen atoms in liquid nitriding is a thermal activation process. Its spread to satisfy the following equation:

$$\frac{d\xi^2}{dt} = A_0 g \exp(-\frac{Q}{RT} g) \tag{1-1}$$

ξ is the nitrided layer thickness, T is the nitriding time, R is the gas constant, take the logarithm on both sides after making integrals:

$$\ln\xi = -\frac{1}{T}\cdot\frac{Q}{2R} + \frac{1}{2}\cdot(\ln A_0 + \ln t) \tag{1-2}$$

It can be seen that A linear relationship with lnξand1/T, and the size of the slope stands for diffusion activation energy. In the aging treatment, aging time: $t=15.552\times10^6$s that is the six months, we can make a test curve about the nitride layer's thickness of the incremental d Lnd and 1/T. Figure5.shows that the calculation of diffusing activation energy of nitride. It can be seen from Figure5.that the experimental data is also basically in a straight line. This result suggests that aging treatment of nitrogen atom diffusion is also in the process of thermal activation process. This can be calculated that diffusing activation energy of nitride is 340.2kJ/mol after six months aging treatment. The value is higher than 304 austenitic stainless steels' (107kJ/mol) which is calculated in the literature [15]. The reason for this result is the stainless steel containing a large amount of alloy elements which hindered the diffusion of nitrogen atoms and reduced the rate in each phase such as γ′and ε. So the permeability rate is reduced.

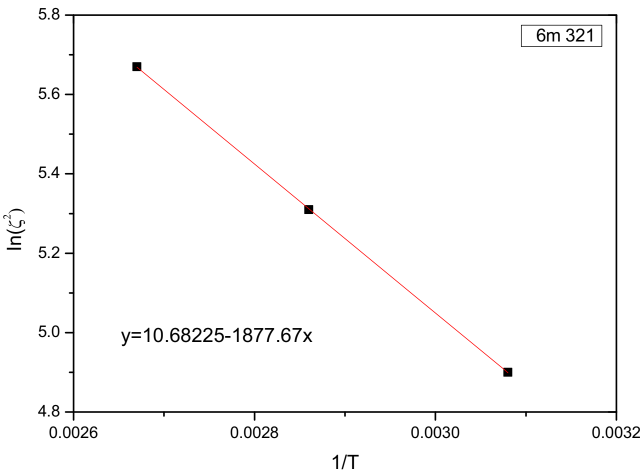

Figure 5. Calculation of diffusing activation energy of nitride.

3.3. Hardness

Figure 6 reveals the relationship between the microhardness of the nitriding layer as a function of aging time. Statistics indicate that the microhardness increase after dealing with salt bath nitriding and first increased and then decreased phenomenon appears in the process of aging treatment. For the nitrided at 430K for 8h and 1.2096×10^6s aging sample, the hardness of the sample increased about 4 times compared with that of the substrate. The highest hardness value obtained in this experiment is about 1430 Hv0.098.This special structure is due to the intrusion of nitrogen and carbon atoms during nitriding at 430K. Twinning is induced by the stress induced by the over saturated nitrogen. The microhardness reaches very high values can be explained most large misfit dislocations induced by double structure group, a large number of relevant stress field and stacking fault solution supersaturating caused by nitrogen [16-18]. In other words, it is the solution strengthening, the dislocations strengthening, and the precipitates strengthening that lead to hardening of the nitrided layer. The nitride layer thickness was increased with the increased of the aging time. The surface of the nitriding layer is oxidizing slowly. Under the role of driving force, the nitrogen atoms diffuse to the matrix of internal constantly. Diffusion coefficient and diffusion velocity of nitrogen atoms are increasing when the aging time increasing. So the nitrogen content in the layer could be decreased gradually and the microhardness of nitrided layers is lower than untreated, the decreasing speed of the hardness is reduced when the aging time more than 15.552×10^6s. On the one hand, diffusion of the nitrogen atoms reaches equilibrium states nearly. Alloying elements such as Cr, in austenitic stainless steel hinder the diffusion of nitrogen in every phase and then the speed of diffusion is decrease. On the other hand, the enhanced nitrogen migration along grain boundaries is strongly connected with a precipitation of CrN, even at low temperature. Wang [19] also observed that chromium nitride precipitation appeared in the nitrided layer after plasma nitriding for 44 hours at 420°C. But overall, the hardness value decreases with prolonging the aging time. There is no doubt that the sample which is nitrided at 430K for 8h and 20.736×10^6s aging that the microhardness value obtained in this experiment is dropped to below 600 $Hv_{0.098}$.

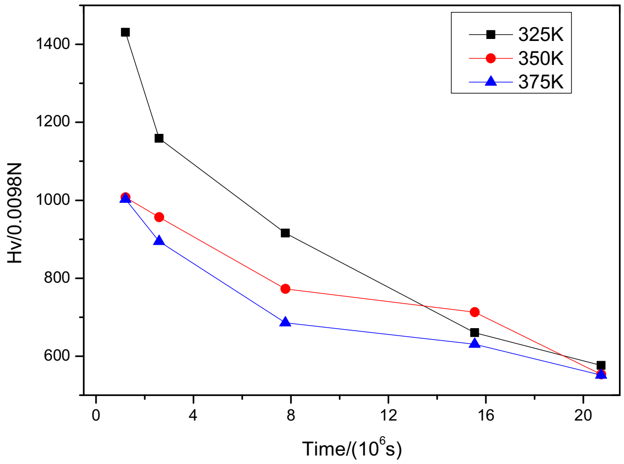

Figure 6. Microhardness of AISI 321 ASS with different aging temperature and time.

3.4. Corrosion behavior

Figure7.shows the typical potentiodynamic polarization curves of the different times obtained in 3.5 mass% NaCl solutions at room temperature for comparison. Icorr, anodic/cathodic Tafel slopes, and corrosion rates measured for samples in different aging times under the potentiodynamic polarization technique are showed in Table 2.

The experiment results revealed the phenomenon that the corrosion rate of the sample is significant different in different aging time and temperature. When nitride for 8 hours, the corrosion rate of the sample is 0.17153mm/a. This is more than the rate of sample which treated for 1.2096×10^6s (0.56181mm/a). The corrosion rate of the sample is decrease slowly with the aging time prolonging. When the aging time at 6 months, the corrosion rate decreases to 0.050165mm/a and the Ecorr is -0.53779V. According to the above analysis, we can draw a conclusion that the proper aging treatment process can obviously improve the corrosion resistance of austenite stainless steel AISI 321 after low-temperature liquid nitriding in the 3.5mass% NaCl solutions.

The beneficial effects of nitrogen in stainless steel have been previously presented in some papers [20, 21]. Some literatures suggests that nitrogen in stainless steel will dissolve during corrosion process and the reaction equation is: [N] + 4H + + 3e- → NH4. This will produce a partial acid corrosion pit in the surface, resulting in corrosion of the growth rate decreased gradually. Kuczynska Wydorska [22] saids that it is one of the important processes that nitrogen atom’s intrusion improves the corrosion resistance of nitrided sample. It is seen that the longer is the aging time; the lower is the corrosion rate. However, the aging time more than 15.552×10^6s, the corrosion current increase, the corrosion voltage decrease and the corrosion rate is also increase slightly. The reason is that after a long period of time, in the S phase generated the CrN second phase material. They create some poor Cr area nearby, reduces the electrode potential, on the other hand, it also can form more micro batteries, accelerating the corrosion in the medium. On the whole, the corrosion property of the nitrided sample through aging treatment is better than untreated sample. Its corrosion resistance is increase to the maximum and then decrease with prolong aging time.

Table 2. Corrosion parameter for various specimens obtained in 3.5% NaCl solution at room temperature by the cycle volt ampere polarization method.

t(aging)/d	ba/mV	bC/mV	ICorr/(A/cm2)	ECorr/V	V(corrosion rate)/(mm/a)
0	179.060	89.095	1.5419E-5	-1.0169	0.56181
14	715.72	122.21	5.0503E-5	-1.0131	0.22222
30	539.47	144.03	1.9974E-5	-0.9861	0.17153
90	431.12	525.11	4.5095E-6	-0.53779	0.050165
180	760.51	399.46	4.6926E-6	-0.57332	0.052201
240	565.239	912.23	1.6177E-5	-0.62029	0.177955

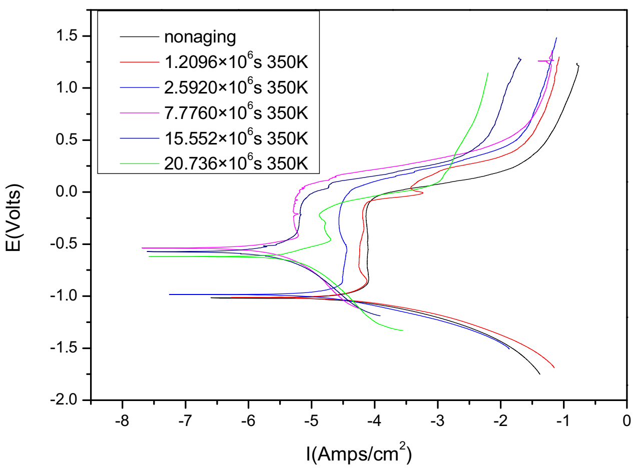

Figure 7. Comparative polarization curves for different times in 3.5 mass % NaCl solutions at room temperature.

3.5. Tensile properties

The tensile properties of AISI 321 ASS of strength treated states with different aging temperature and time are shown in Figure 8. The results indicate that the tensile strength of AISI 321 ASS decreases significantly after salt bath nitriding treatment. The strength value is decrease to 673MPa in 2.592×10^6s at 325K. This phenomenon shows that although the AISI 321 ASS is endowed with the high surface hardness, it is seen that the tensile properties of strength decreases after salt bath nitriding treatment. It can also be known that the strength value fluctuates within a certain range in the long term aging treatment, which is caused by the diffusion of nitrogen atoms.

Figure 9 shows the changes of elongation of the 321 stainless steel with different aging temperature and time after salt bath nitriding treatment. It is clearly that elongation value of AISI 321 ASS increase compared with the non-nitrided samples. What's more, the elongation value still increased with the increase of aging time and aging temperature. But more than 15.552×10^6s, the elongation value began to decline. It can be indicated that the liquid nitriding and aging treatment can effectively improve the elongation of the 321 stainless steel. Considering Fick's second law, the weak decrease elongation should be attributed to the diffusion of nitrogen atoms in large quantities form the nitriding layer. So after 20.736×10^6s at 375K, elongation has dropped from 70.9% to less than 65%.

Stress strain curve for different aging temperature in six months are shown in Figure10 for comparison. It also can be seen in Figure10 that the tensile strength decreases a little compared with the untreated sample. The experiment results showed that there is an obviously difference in different tensile properties in different aging treatment. In the process of aging, the tensile strength still has a decline in different degrees (Figure10) .But the change of the plastic is opposite. The elongation slowly improves owing to the liquid nitriding and aging treatment. For example, in 15.552×10^6s aging time, the elongation value increased from 69% to 70.2% with the increase of aging temperature from 325K to 375K. The rising values of the elongation observed can be explained by large to the diffusion of nitrogen atoms related with supersaturation of nitrogen in austenite and fine dispersed the second phase precipitates subsequently.

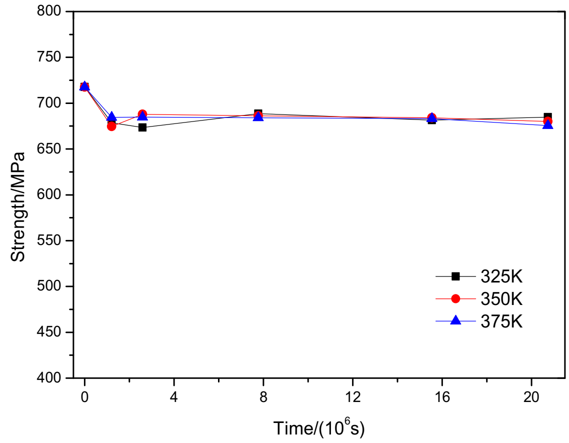

Figure 8. Tensile strength of low temperature salt bath nitrided AISI 321 ASS changes with different aging temperature and time.

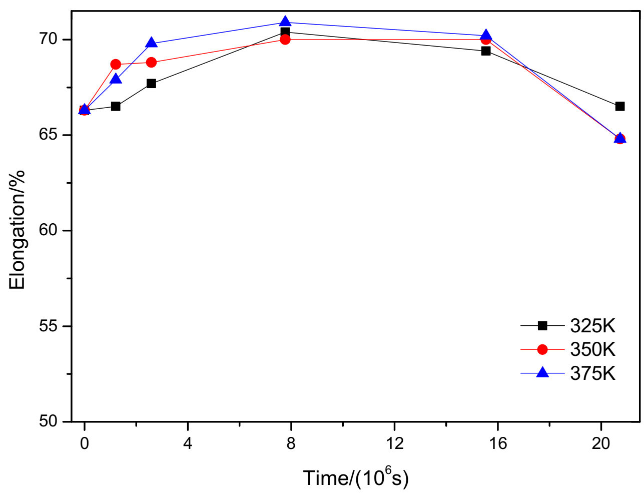

Figure 9. Elongation of low temperature salt bath nitrided AISI 321 ASS changes with different aging temperature and time.

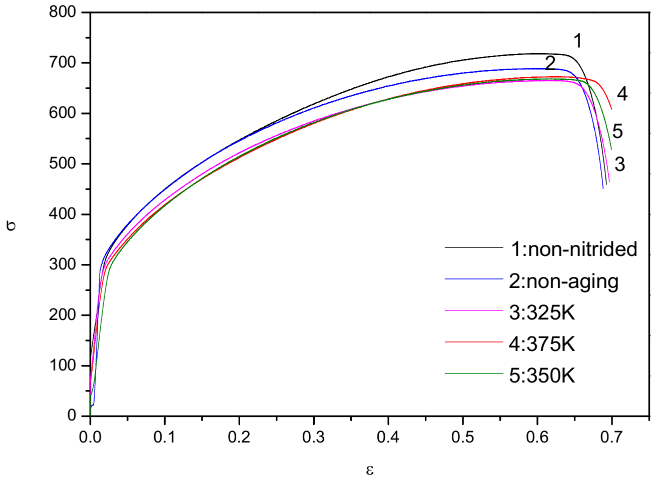

Figure 10. Stress strain curve for different aging temperature in15.552×106s.

4. Conclusion

When stainless steel AISI 321 is dealing with salt bath nitriding at 430K, the main phase of the nitride coating layer is the expanded austenite (S phase). But with the change of the aging treatment, microstructural morphology of nitriding layer has obvious difference. The thickness of the expanded austenitic nitriding layer formed on the surface of the sample is in the range of 7.6 to 22.5 μm. The nitride layer is growth owing to nitrogen diffusion which is according to the expected parabolic rate law. Some mechanical properties such as the surface microhardness and the plastic improve effectively when the aging treatment time and temperature prolonged to proper parameter. However, the tensile properties of AISI 321 ASS of strength decrease after salt bathing nitriding and aging. The proper aging treatment also can improve the corrosion resistance.

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