Effect of Aging on Stress Relaxation of Inconel X-750 Bolt at 923 and 1033 K

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ABSTRACT

The effect of aging temperatures on stress relaxation of Inconel X-750 bolt at 923 K and 1,033 K for 1,800, 3,600, 5,400 and 7,200 ks was investigated. The samples were solution heat treated at 1158 K for 86.4 ks and aging at 953, 973, 993, 1,013 and 1,033 K for 72 ks. The German SEP test guideline was followed for the stress relaxation test. The initial stress on bolt was 450 MPa. The hardness and tensile strength of samples were also investigated at room temperature after stress relaxation tests. Based on our previous work, aging at those temperatures did not provide any change in the average diameter of austenite grain. However, the average diameters of gamma prime precipitated in austenite matrix were in between 10-33 nm. The results showed that, by aging at 953 K and 973 K for 72 ks, the samples provided the best resistance to stress relaxation at 1,033 K and 923 K, respectively. The lowest stress relaxations at 923 K and 1,033 K were 22 and 33%, respectively. The hardness testing result agreed well with the result of tensile strength testing. For stress relaxation tests at 923 K, the maximum hardness and tensile strength were obtained with optimum size of precipitated particles at relaxation time in the range of 1,800-3,600 ks for all aging temperatures. However, for the stress relaxation tests at 1,033 K, the hardness and tensile strength decreased for all aging temperatures due to rapid coarsening of gamma prime particles.

Keywords: Aging, Stress relaxation, Inconel X-750, Bolt

1. INTRODUCTION

It is well known that nickel base, nickel-iron base and cobalt base super alloys are materials using at high temperature, because they perform very good creep, oxidation and corrosion resistances /1-3/. These materials are applied for bolt, rotor blade, wheel, disc, etc, for jet engine or gas turbine components. Generally, wrought superalloys, e.g., Inconel X-750, Inconel 718, IN792, Nimonic 80A, and A286, are the most selected alloys for those components /4-7/. One of the problems in using superalloy at high temperatures is creep or stress relaxation. These behaviors are influenced by microstructure changes during service /1-5/. With an

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appropriated microstructure design by heat treatment and aging, the possible long service life of superalloy regarding stress relaxation can be achieved. The stress and sizing designs of gas turbine components made of superalloy need stress relaxation data of the heat-treated superalloy /4, 5/. However, there are a few stress relaxation data of some superalloys found in the literature /4, 5/. In this work, the effect of aging temperatures after solution heat treatment on stress relaxation of Inconel X-750 bolt for gas turbine component at 923 K and 1023 K for 1,800, 3,600, 5,400 and 7,200 ks was investigated. The microstructure and mechanical properties of Inconel X-750 bolt in as received state, after solution heat treatment and aging were ready investigated and reported in our previous work /8/.

2. EXPERIMENTAL PROCEDURE

The material after annealing was supplied by Aigle High-Temp Product Co. Ltd. The average chemical composition by weight percentage of the tested materials is showed in Table 1. It shows that the tested material was Inconel X-750 according to AMS 5667. The stress relaxation test followed the German SEP 1260 test guideline /9/. Bolt, sleeve and nut were prepared from the test material. Figure 1a-c shows their drawing detail. They were firstly solution heat treated at 1,158 K for 86.4 ks, then rapid cooled in air. Bolts were aged at 953, 973, 993, 1,013, and 1,033 K for 72 ks. It should be noted that sleeves and nuts were usually used as the support parts; then they were generally aged at 973 K for 72 ks, which is the standard aging condition of Inconel X-750 /1/. After that, the test specimens were set up from bolt, sleeve and nut. The specimen example is shown in Fig. 1d. Stress relaxation tests were done at 923 and 1023 K for 1,800, 3,600, 5,400 and 7,200 ks. Table 2 summarizes the conditions of solution heat treatment (SHT) and aging (A) for bolt and those of the stress relaxation test (SRT) for specimens. Figure 2 shows the schematic plot for study the stress relaxation of specimens described in Refs. /4,10/. The initial stress of all bolts was set at 450 MPa for every test condition. The Vickers hardness and tensile test (ASTM E8M-94) of bolt after stress relaxation test were determined. The microstructures of bolt in as received state, after solution heat treatment and aging were also studied using TEM. For TEM observation, the specimens were electrolytic etched in a solution of 133-cm' Acetic acid + 25 g of CrO_3 + 7 cm^3 of H_2O at 10-12 V, and 293-298 K /11/.

Table 1

Chemical composition (mass %) of the test material and material specification according to ASM 5667 by emission spectroscopy

Material	Composition (mass %)									
	Ni	Cr	Fe	Ti	Nb	Al	Co	Cu	C	Si
Test Material	73.07	14.71	6.902	2.609	0.991	0.662	0.897	0.075	0.044	0.08
Inconel X-750 (ASM 5667)	Min. 70.00	14.00-17.00	5.00-9.00	2.25-2.75	0.70-1.20	0.40-1.00	1.00	0.50	0.08	0.50

Table 2

Summary of the conditions for heat treatment and stress relaxation test

Items	Temperature (K)	Time (ks)	Cooling
Solution heat treatment (SHT)	1,158	86.4	Air
Aging (A)	953, 973, 993, 1,013, 1,033	72	Air
Stress relaxation test (SRT)	923, 1,033	1,800, 3,600, 5,400, 7,200	Air

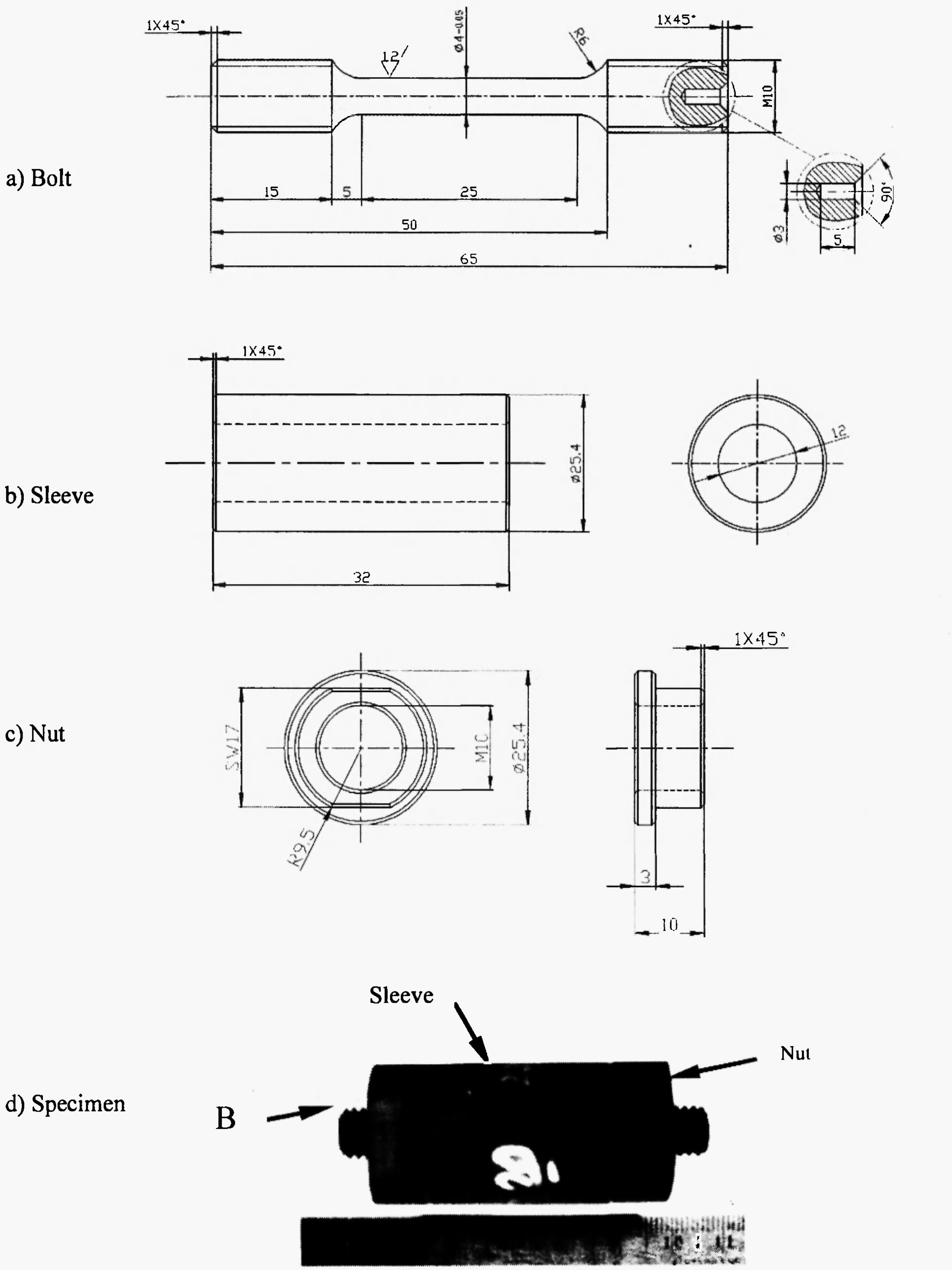


Fig. 1: Drawing detail of bolt, sleeve, nut and example of specimen

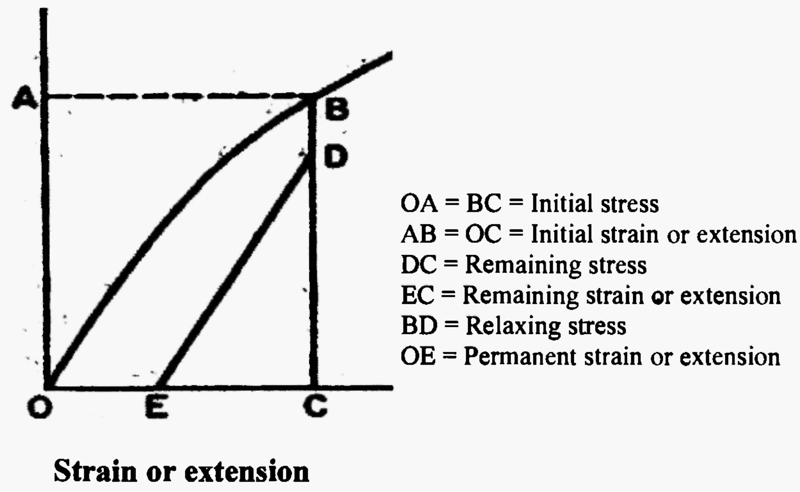


Fig. 2: Schematic plot of stress-strain or stress-extension for stress relaxation evaluation [4, 7]

3. RESULTS AND DISCUSSION

The observed microstructures of bolt under optical microscope, SEM and TEM have already been reported /8/. Here only the observed microstructure related to the discussion is shown. To explain the results, the abbreviated terms to describe the heat treatment condition and stress relaxation test are used. For example, "SHT 1,158, 86.4 ks + A 973, 72 ks" means "solution heat treatment at 1,158 K for 86.4 ks and aging at 973 K for 72 ks" and "SRT 923" means "stress relaxation test at 923 K". The remaining stresses or stress relaxations of specimens were evaluated from stress-extension curves. Figure 3 shows the example of stress-extension curve of bolt (SHT 1,158, 86.4 ks + A 973, 72 ks) before and after SRT 923 for 3,600 ks. All other similar stress-extension curves of all other tests were demonstrated elsewhere /12/. It was clear that the initial stress of 450 MPa loaded on bolt was reduced to a level after long term heating. Figures 4 and 5 show the remaining stresses on bolt in relation with relaxation times at 923 K and 1,033 K, respectively. The introduction of the hold time at tensile strain allowed for the conversion of elastic to plastic strain, i.e., stress relaxation occurred. The relaxation of remaining or residual stresses might be determined by diffusion of dissolved alloying elements which are homogeneously distributed and by microstructural mechanisms according to previous work /13/. The remaining stresses have been reduced due to the mechanisms such as diffusion-controlled rearrangements of dislocation structure and alterations of precipitation configurations, respectively. Both processes are often linked with each other. This could occur, especially, at threads and contact faces leading to the loss of pre-load. This is a substantial difference compared to the relaxation test in the relaxation machine with smooth specimens /13/. The stresses reduced abruptly in the range of 0-1,800 ks and kept nearly constant at some levels during 3,600-7,200 ks in both tested temperatures. It seems that the lower aging temperatures resulted in the higher remaining stresses, i.e., the less stress relaxation. In SRT 923 and SRT 1,033, the highest remaining stress was obtained, when aging conditions were A 973, 72 ks and A 953, 72 ks, respectively. The recommended aging temperature is 973 K for this nickel base super alloy /1/. It should be noted that the higher temperature in stress relaxation testing provided the lower residual or remaining stress at the same relaxation time of each heat treated condition, due to the decrease in modulus of elasticity as temperature rose, as well as the higher rate of diffusion mechanism. The results may imply that when the alloy is used at temperatures somewhat lower than 973 K, the appropriate aging temperature should be 973 K. This may be related to the mechanism of dislocation movement over the gamma prime (γ') particles with the

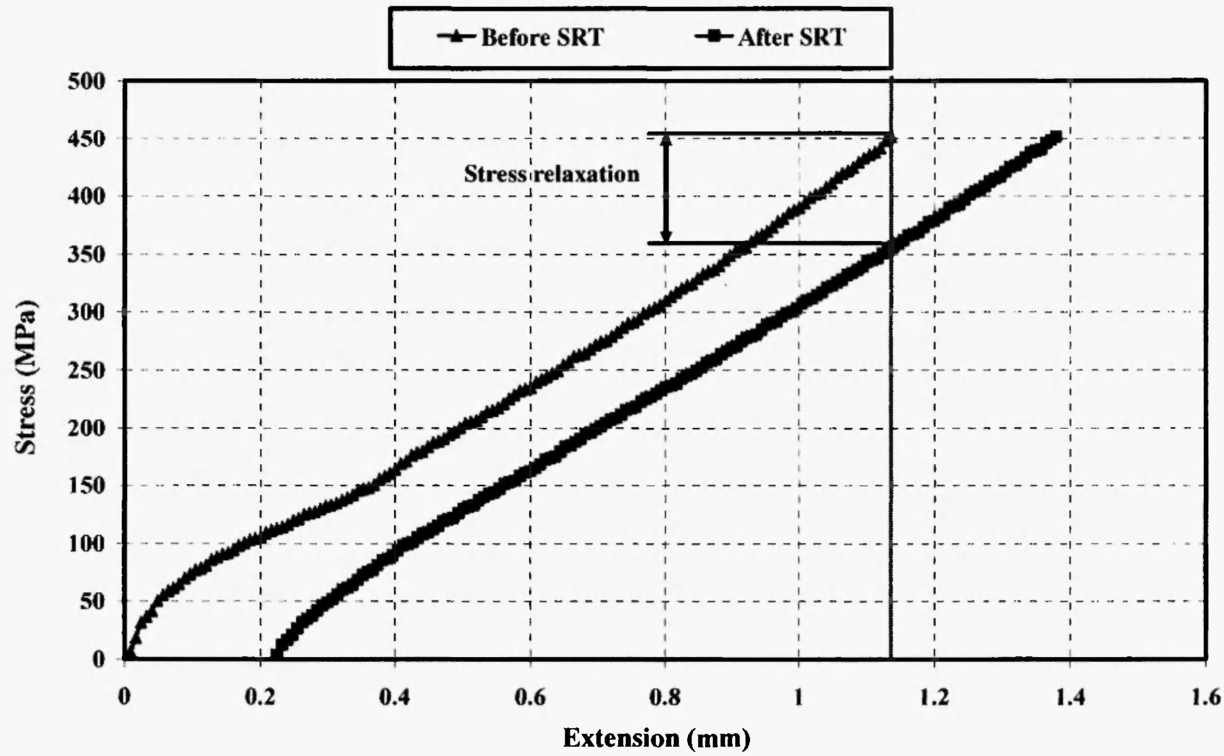


Fig. 3: Example of stress-extension curve of bolt (SHT 1158 K, 86.4 ks + A 973 K, 72 ks) before and after stress relaxation test at 923 K for 3600 ks

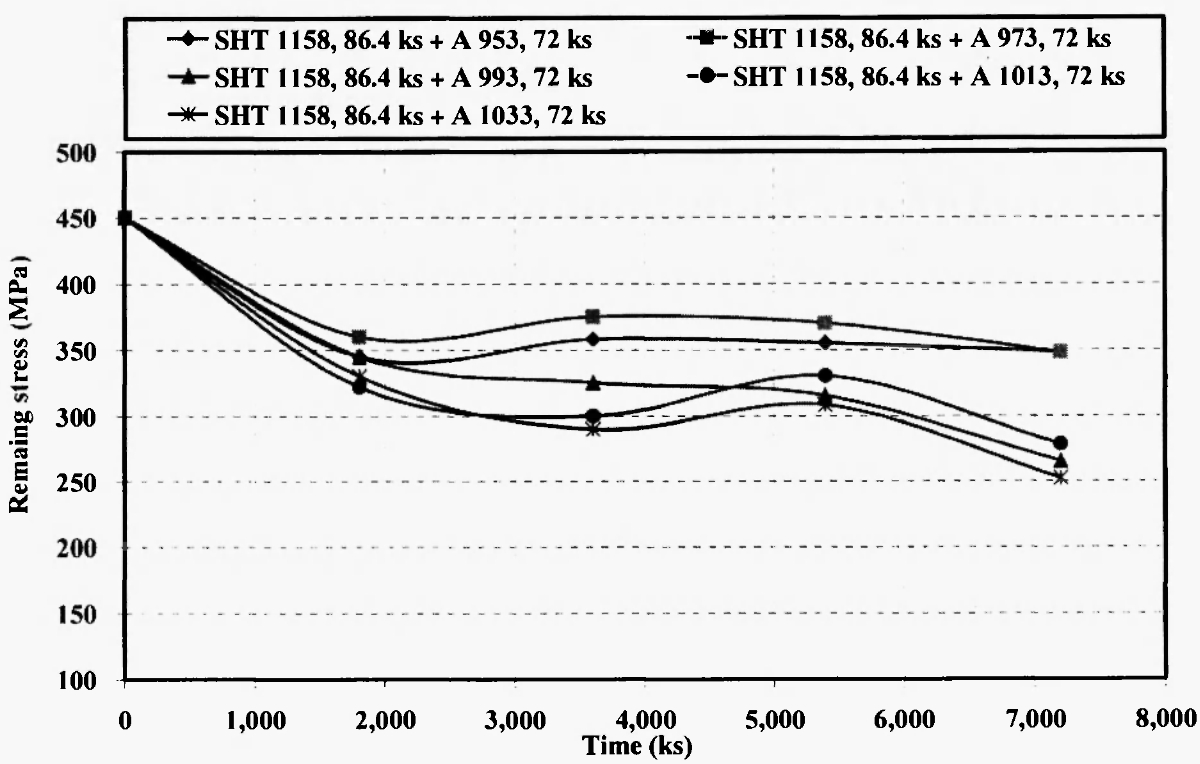


Fig. 4: Remaining stress of bolt and relaxation time at 923 K

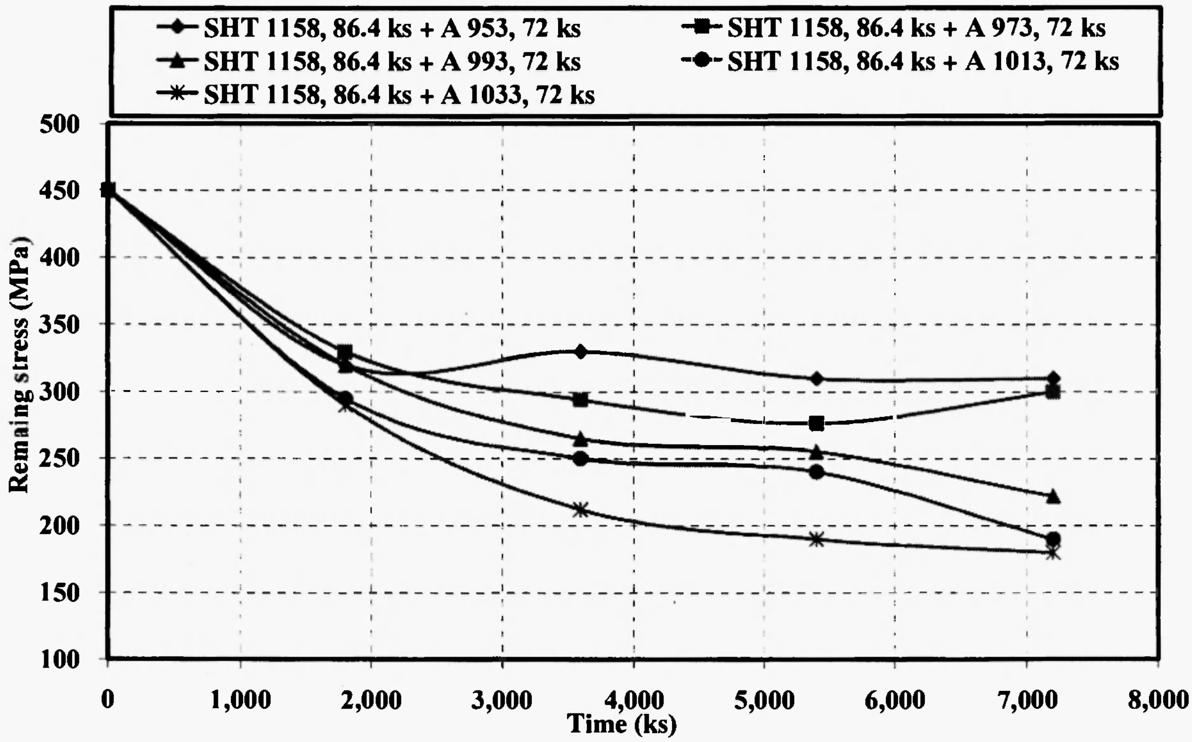
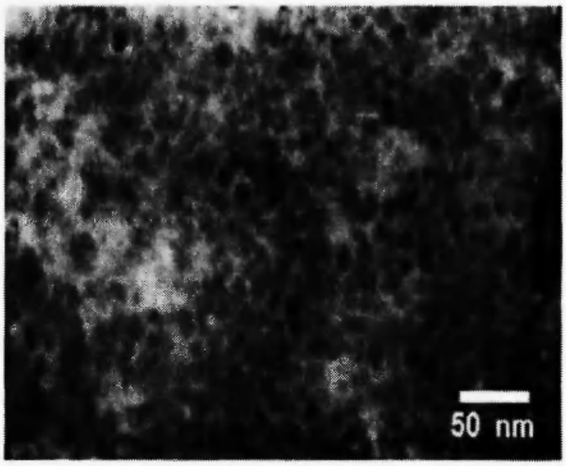


Fig. 5: Remaining stress of bolt and relaxation time at 1033 K

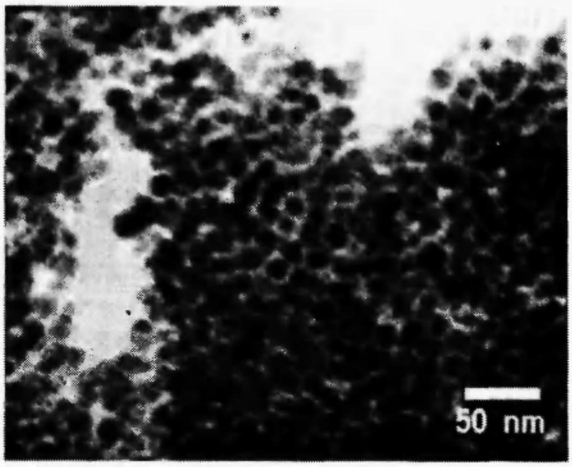
proper initial size, which precipitated in the austenite (γ) matrix. It should be remarked that at constant temperature of isothermal relaxation, the coarse precipitates present, especially at higher stress relaxation testing temperature of 1,033 K with faster rate of γ′ coarsening, would be expected to reduce creep strength since they will not be an optimal size and distribution to impair dislocation movement. The existence of coarse precipitate in new components resulted in reduced short-term performance and would be also contribute to rapid aging under relaxation test /14/. It should be noted that, in this study, the effect of different dependent anelastic strain on grain size would not be considered due to the same value of initial and after testing grain size in each aging condition.

From our previous work, increasing aging temperatures ranging from 953-1,033 K for 72 ks resulted in increasing initial size of gamma prime particles, which are in the range of 10-33 nm /8/. Figure 6 shows the example TEM micrographs of gamma prime particles after aging. Dislocations may pass the smaller gamma prime particles by shearing mechanism but pass the larger gamma prime particle by bypassing or bowing mechanism /1/. From Figures 4 and 5, the minimum stress relaxations of 22 and 33% were obtained at SRT 923 and 1033, when the bolts were aged at 973 and 953 K, respectively.

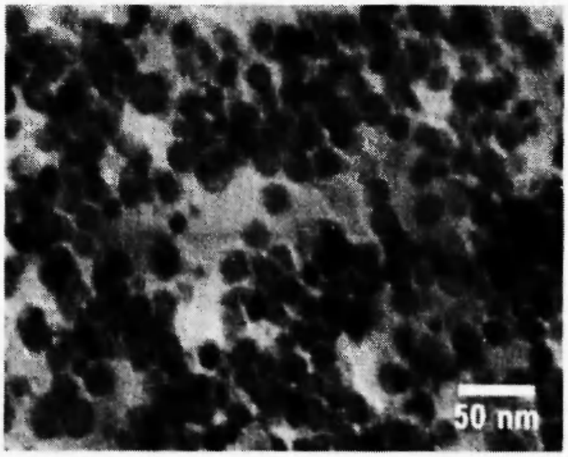
The hardness of bolt measured after various relaxation times was showed in Figs. 7 and 8 for SRT 923 and SRT 1,033, respectively. For SRT 923, the hardness increased to the maximum at relaxation times around 1,800-3,600 ks and then all decreased to a steady level after 3,600 ks. For SRT 1,033, the hardness decreased continuously and rapidly for all aging temperatures till 5,400 ks, then seemed to be constant for longer SRT time. These results were coincident with the tensile test results shown in Figs 9 and 10. For SRT 923, the tensile strength increased to a maximum at relaxation times around 1,800-3,600 ks and after that decreased for all aging temperatures. For SRT 1,033, the tensile strength decreased for all aging temperatures. According to the principle of precipitation hardening /15/, it is well known that when the alloy is underaged, the strength and hardness increase. They reach the maximum at the peak age and decrease when the alloy is over aged. Considering the gamma prime particle size



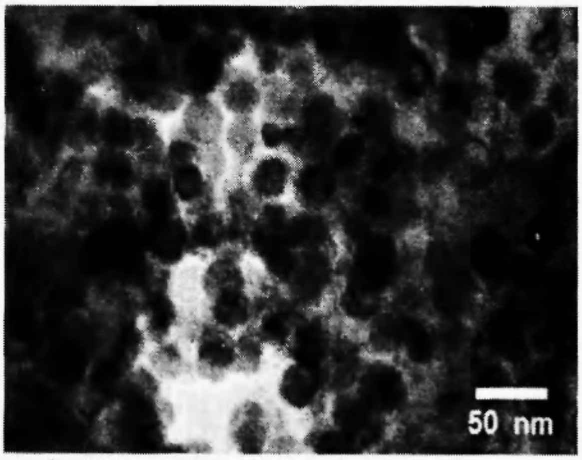
a) SHT 1158 (86.4) + A 953 (72)



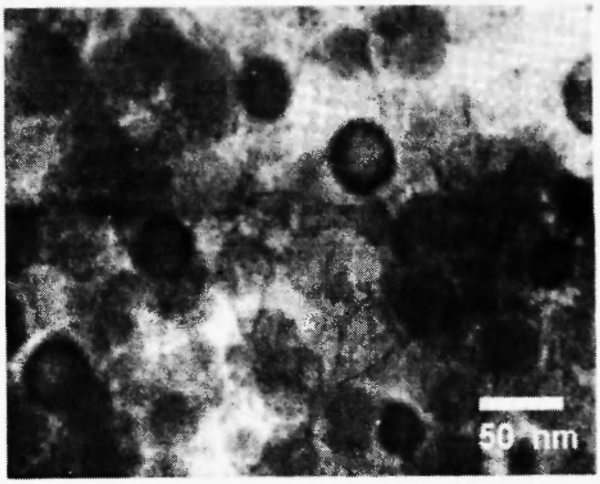
b) SHT 1158 (86.4) + A 973 (72)



c) SHT 1158 (86.4) + A 993 (72)



d) SHT 1158 (86.4) + A 1013 (72)



e) SHT 1158 (86.4) + A 1033 (72)

Fig. 6: TEM micrograph of gamma prime precipitated in austenite matrix after various solution heat treatments and agings

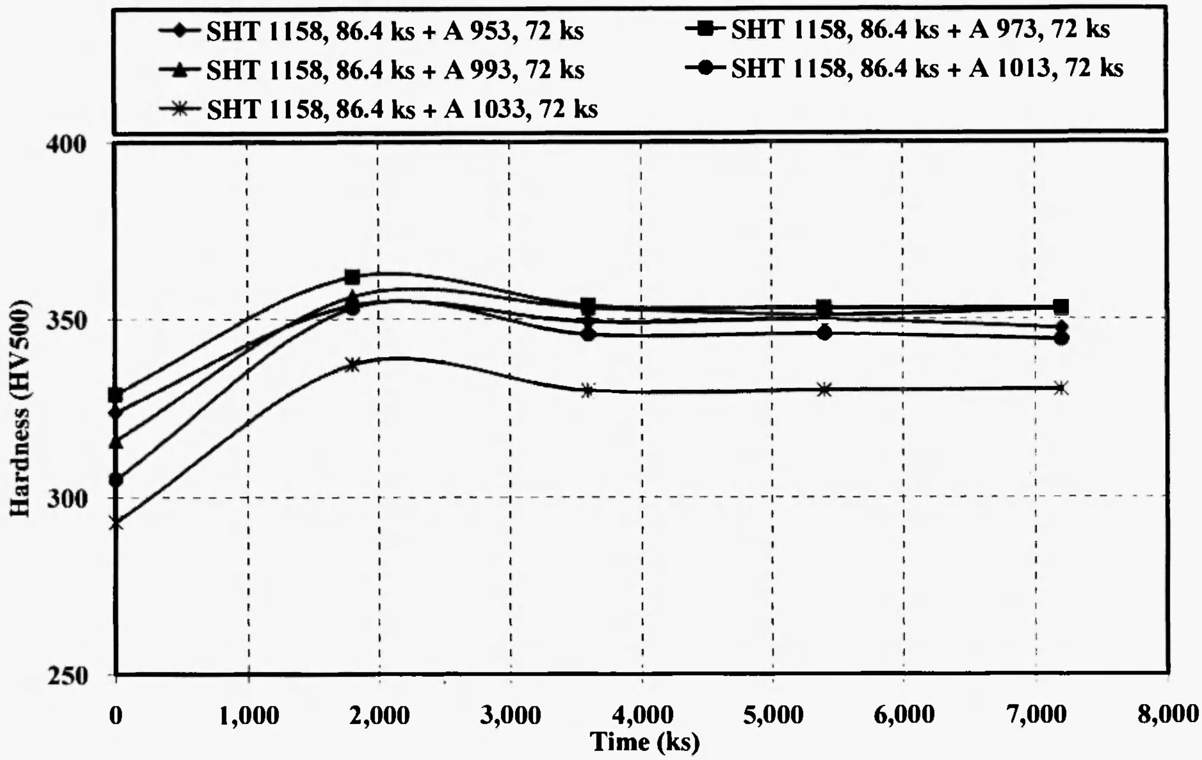


Fig. 7: Hardness of bolt and relaxation time at 923 K

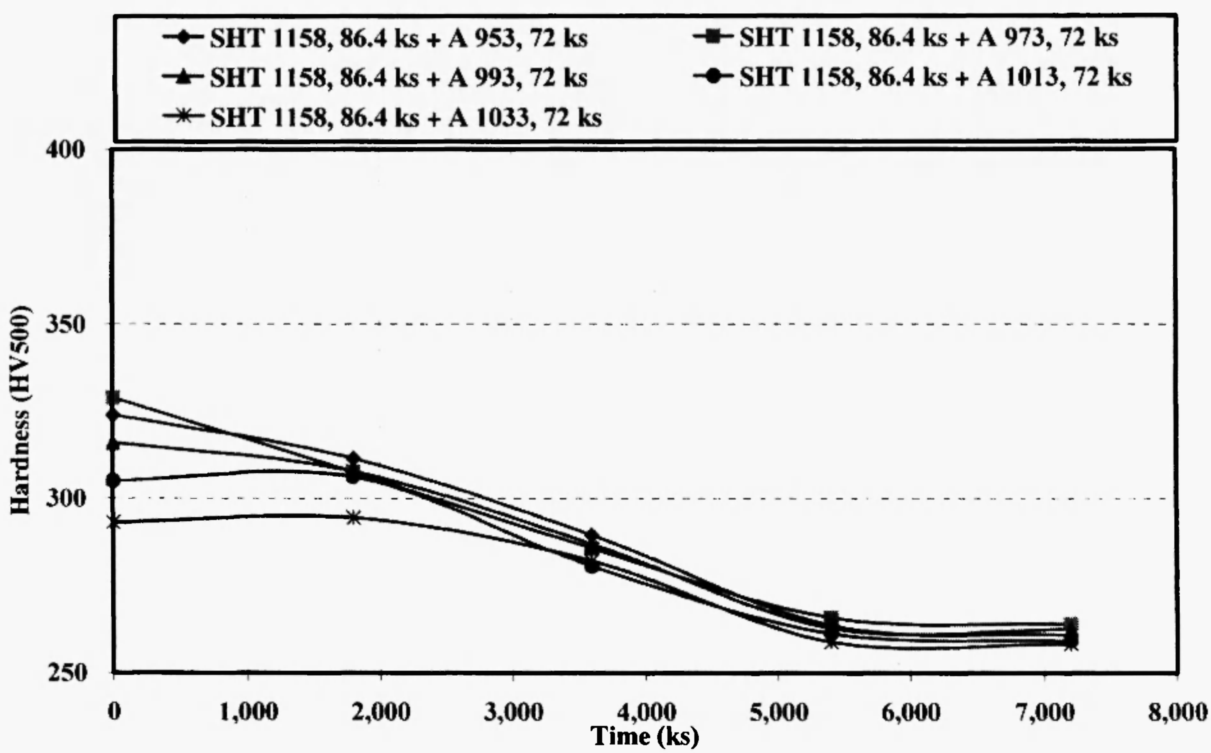


Fig. 8: Hardness of bolt and relaxation time at 1033 K

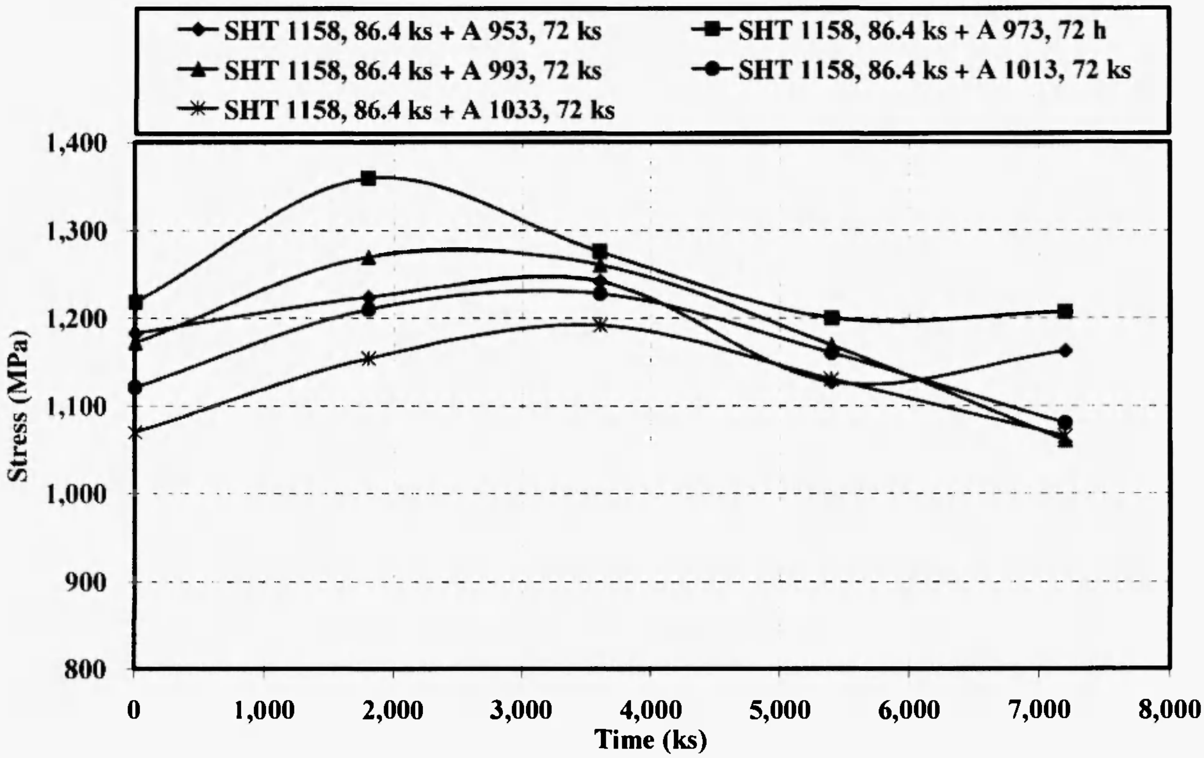


Fig. 9: Tensile stress of bolt and relaxation time at 923 K

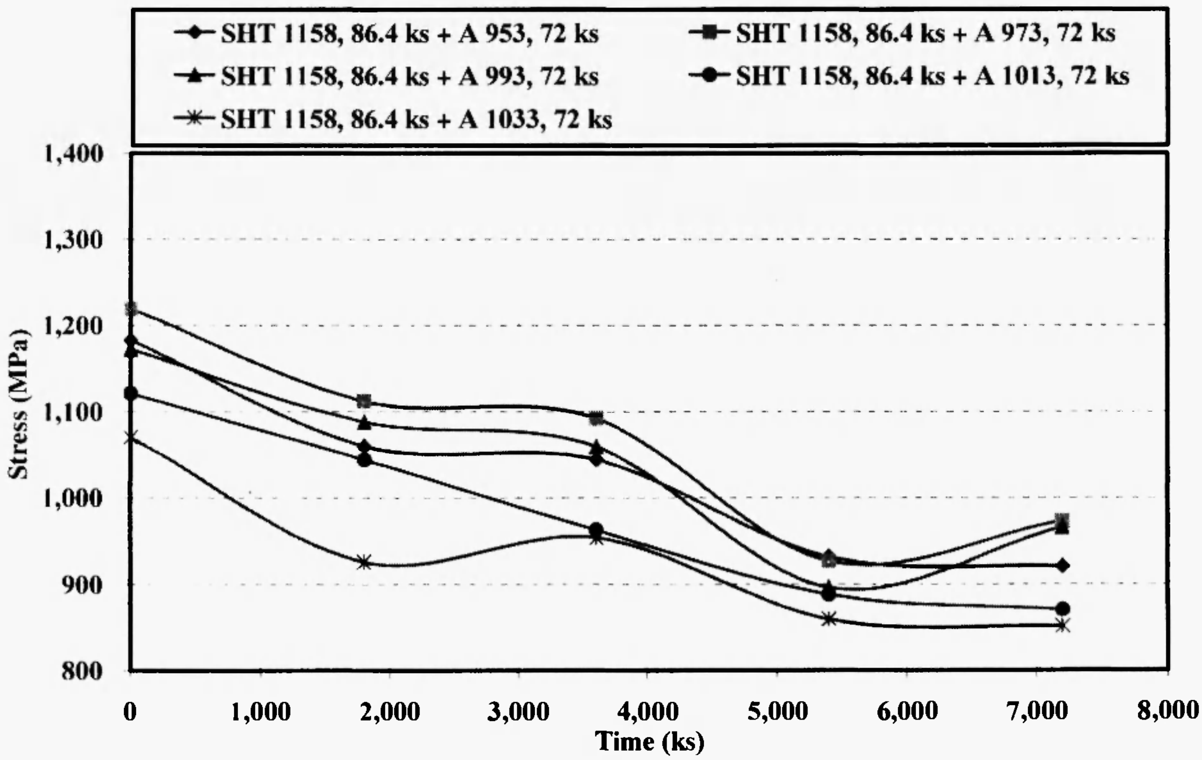


Fig. 10: Tensile stress of bolt and relaxation time at 1033 K

and state of aging, it is possibly said that the size of < 10 nm is in the aging state, ~15 nm is at the peak age state, and 20-40 nm or over is in the overaging state /16/. From our previous work /8/, by aging at 953 K, the average gamma prime particle size is 10 nm; hence, it was underaged state and by aging at 1,033 K, the average size is 33 nm, hence, it was overaged state.

The SRT 923 of Inconel X750 after SHT 1,158 + A 953-1,033 may be still in the state of "underaging" in the period of 0-1,800 ks, in the state of "peak aging" at around 1,800-3,600 ks and "overaging" after 3,600 ks. Therefore, the maximum hardness and strength results were archived. The SRT 1,033 of Inconel X-750 after SHT 1,158 + A 953-1,033 may be in the state of "overaging". Therefore, the hardness and strength continuously decreased when the relaxation time increased. The growth rate of gamma prime at 1,033 K is faster than that at 923 K. This may also accelerate the decrease of hardness and tensile strength for SRT 1,033.

4. CONCLUSIONS

In this work, the effects of aging temperatures at 953, 973, 993, 1,013 and 1,033 K, 72 ks, after solution heat treatment at 1,158 K, 86.4 ks, on stress relaxation behavior of Inconel X-750 bolt at 923 K and 1033 K for 1,800, 3,600, 5,400 and 7,200 ks were examined. The samples performed the highest resistance to stress relaxation at 923 K and 1,033 K by aging at 973 K and 953 K for 72 ks, respectively. The lowest stress relaxations at 1,033 K and 923 K were 33 and 22%, respectively. For stress relaxation at 923 K, the maximum hardness and tensile strength were obtained at the stress relaxation time of 1,800-3,600 ks for all aging temperatures. However, for stress relaxation at 1,033 K the hardness and tensile strength decreased.

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