Effect of Ultrasonic Nanocrystal Surface Modification to the Fatigue Properties of S45C with Different Surface Hardness

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Abstract The Ultrasonic nanocrystal surface modification (UNSM) was employed to enhance the fatigue strength of S45C steel with different pre-plasma nitriding (un-nitrided, nitriding 8 h and 48 h) by improving the surface roughness and producing nanocrystalline on the sample surface. The parameters of UNSM were two different strike numbers, 34000 mm^{-2} and 68000 mm^{-2}. Different experimental processes including microstructure observation, microhardness, roughness and X-ray diffraction measurements have been performed to characterize the treated surface of specimens. The results shown that after UNSM treatment an improvement can be obtained for surface microhardness, surface roughness, and compressive residual stress. Under the same UNSM conditions more surface defects can be produced with the increasing strike number and with increase of surface hardness the effect to surface roughness decreased. For the higher hardness surface sample, smaller nanocrystalline can be obtained compared with other sample through XRD results. Higher compressive residual stress was found for un-nitrided S45C steel with increasing strike number, but a decrease was found for the nitriding sample. Rotating bending fatigue test was carried out to evaluate the effect UNSM to the fatigue strength. An improvement of fatigue strength for un-nitrided and nitriding 8 h sample was found and the surface defects were the main reason inducing the fatigue failure. However, for N48 samples, no improvement of fatigue strength as little effect of UNSM on the sub-surface crack induced by inclusion.

Keywords UNSM, Plasma nitriding, Surface Hardening, Fatigue

1. Introduction

Ultrasonic nanocrystal surface modification (UNSM), as one of severe surface plastic deformation (S^2PD) methods to improve material surface properties, has been used on many materials treatments [1-5]. The properties of wear [4, 5], fatigue [1-3] of materials can be improved as the reason of high surface hardness and compressive residual stress produced by the UNSM treatment. In the study of Cao [2], with the increase of strike number higher fatigue strength can be obtain and small fatigue crack can be restrained as the compressive residual stress produced by UNSM treatment. Suh's study [3] shows that the grain in the top surface zone can be refined into nano-size and the depth of gradual grain refined layer about 100 μm can be produced by UNSM treatment with the applied static force of 100 N. Amanov [5] has found that high impact load can get a lower friction and well wear characteristics and smaller nano-grain size can be obtained at the same time. So we can see that the processing parameters (the applied static force, the strike number per mm^{-2}, the size of tip ball) can be controlled accurately and all of these parameters have a close connection with the characters of specimen's surface zone after the UNSM treatment. Also the structure of the material receiving surface treatment plays an important role for the formation of microstructure in the surface processing layer. Zhou [6] showed that the SMAT steel sample has a thicker refined layer (with grain size < 100 nm) compared with pure Fe. Cho [7] and Lee [8] found that with more hard

phase smaller sized grain can be induced due to the easier formation of dislocation around the hard phase.

Although a few studies have been conducted on the UNSM treatment with many kinds of materials, few studies about the UNSMed sample with different surface hardness were found. In this paper, the specimens of S45C steel with different surface hardness introduced by pre-surface treatments were processed by the UNSM treatment under the same conditions. The surface morphology, microstructure, residual stress, microhardness and fatigue properties were investigated and the effect of different pre-hardening surface to the UNSM treatment on the surface properties was discussed.

2. Experiment

2.1. Materials preparation

The material used was a medium-carbon steel S45C shaft with a chemical composition (in wt.%) of 0.45%C, 0.15%~0.35%Si, 0.6~0.9%Mn, ≤0.03%P, ≤0.035%S, ≤0.3%Cu, ≤0.2%Ni, and ≤0.2%Cr, balanced with Fe. To test the fatigue properties, the specimens were machined to the dimensions shown in Fig. 1, resulting in a stress concentration factor, K_t, of approximately 1.08. The specimens were first quenched (austenitized, 1113 K, 150 min; oil quenched, room temperature, 60 HRC) and tempered (tempered, 773 K, 270 min, 31 HRC). The specimens were then polished by using sandpaper from grade 400 to grade 2000. Subsequently, the specimens were nitrided in a mixture gas (40%N_2-60%H_2) and at 400 Pa. Two nitriding time durations, 8 h (N8) and 48 h (N48), were used to obtain different nitriding properties.

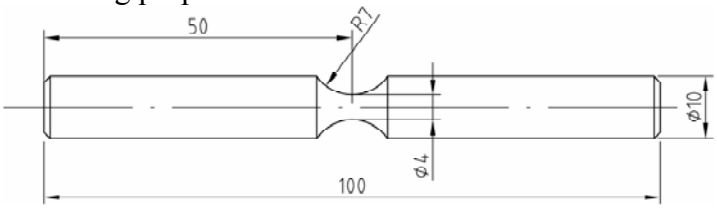

Figure 1. Dimension of the test specimen for S45C steel.

2.2. Surface treatment

After plasma nitriding, the surface layer (approximately 12 μm) was removed to avoid the effect of the nitriding layer on the subsequent UNSM treatment. The UNSM technique details have been previously published. An ultrasonic generator (a 30 μm amplitude and 20 kHz) and air compressor devices were used to produce a static force with a dynamic load to strike the material surface with a tungsten carbide (WC) ball (the diameter: 2.4 mm). Accurate surface strike numbers can be obtained by calculating the speed of the surface being treated when the specimen is rotated by a turning lathe. Thus, it is easy to process the material surface and control the static loads and strike numbers. Throughout the process, cold liquid was used to avoid high specimen surface temperatures.

Table 1. Specimen treatment conditions

Specimens	Untreated	U1	U2	N8	N8U1	N8U2	N48	N48U1	N48U2
34000 mm^{-2}	–	Y	–	–	Y	–	–	Y	–
68000 mm^{-2}	–	–	Y	–	–	Y	–	–	Y

To study the effect of strike number on the fatigue properties of the nitrided sample, two treating

parameters were selected: (U1) strike number of 34000 mm^{-2}; static load of 50 N; revolution rate of 37 rpm, feed rate of 0.07 mm/rev; and (U2) strike number of 68000 mm^{-2}; static load of 50 N; revolution rate of 18.5 rpm, feed rate of 0.07 mm/rev. The specimen processing conditions are listed in Table 1.

2.3. Materials characterization

To examine the grain-refined layer and the nitriding layer, the cross-section of the all specimens were observed by an optical microscope (Olympus BN2). Before test, the samples were mechanically polished with the sandpapers (from grade 150 to grade 2000) and then a polish cloth with a liquid suspension of alumina. The saturated picric acid solution was used to etch the specimens in this test.

X-ray diffraction (XRD) was employed to measure residual stress of all sample varieties, utilizing Cr-Kα radiation for a longer wavelength and deeper penetrating power. The test was carried out on a Rigaku XG-4026A1 using the classical sin2ψ method. Cu-Kα (on a Rigaku Rint-2000) was used to analyze the state of the surface layer nanostructure and the compound layer of nitrided samples to a depth of approximately 5 μm.

The microhardness of the plastic deformation zone and nitriding layer was measured on a micro-Vickers hardness tester (MVK-E3, Akashi). The parameters used in the microhardness test were 50 gf and a duration 15 s.

The surface roughness of the S45C specimens before and after the UNSM treatment was tested on a contact surface profiler (ULVAC DEKTAK3).

To compare the fatigue properties of S45C with the two process methods, a rotating bending fatigue test was carried out on a dual-spindle rotating bending fatigue test machine under atmospheric conditions. The rotating frequency used was 52.5 Hz, and the stress ratio was -1.

The failure fracturing of the samples was observed using scanning electron microscopy (SEM) (S-4700, Hitachi). Energy-dispersive X-ray spectroscopy (EDX), with the same machine, was used to detect the composition of any inclusions that induced the sub-surface fish-eye crack initiation.

3. Results and discussion

The surface morphology of before and after UNSMed specimens was shown in Figure 2. It is obvious that UNSMed processing marks were produced on the specimen surface. As the processing principle, these marks show parallel and as the increase of processing number these marks become more obvious. For the parallel marks, the distance between each other was approximately 70 μm. This value equals to the processing parameter of the feed rate which is 0.07 mm/rev. During surface treatment, only the surface under the center of the tip can receive most severe plastic deformation as the reason of ball shape of the process tip. Comparing the surface morphology results of UNSM samples with different pre-surface hardness, it can be seen that for the softest surface sample (un-treated) the UNSM process marks show the most obvious after the process of 68000 mm^{-2}. The surface roughness was test and shown in Figure 3. It can be found that the surface roughness (Ra) for the all UNSMed sample has an improvement and even there are a few of obvious UNSMed process marks on the UNSMed sample surface. As the increase of strike number, a little growth can be observed with the surface roughness. Combined with results of surface morphology it is easy to find that the UNSMed process marks produced during surface process were the main reason.

Though from magnified surface morphology results the region between two UNSMed marks was smoother the process of more strike number, the UNSMed marks mainly lead to the worse of the surface roughness. For the nitriding samples, the UNSMed marks show unobvious as the reason of nitrides within top surface region reducing the plastic deformation of sample surface. Compared with the soft un-nitriding sample, better surface roughness can be obtained for the sample surface with higher hardness. It also means fewer surface defects produced on the nitriding sample surface.

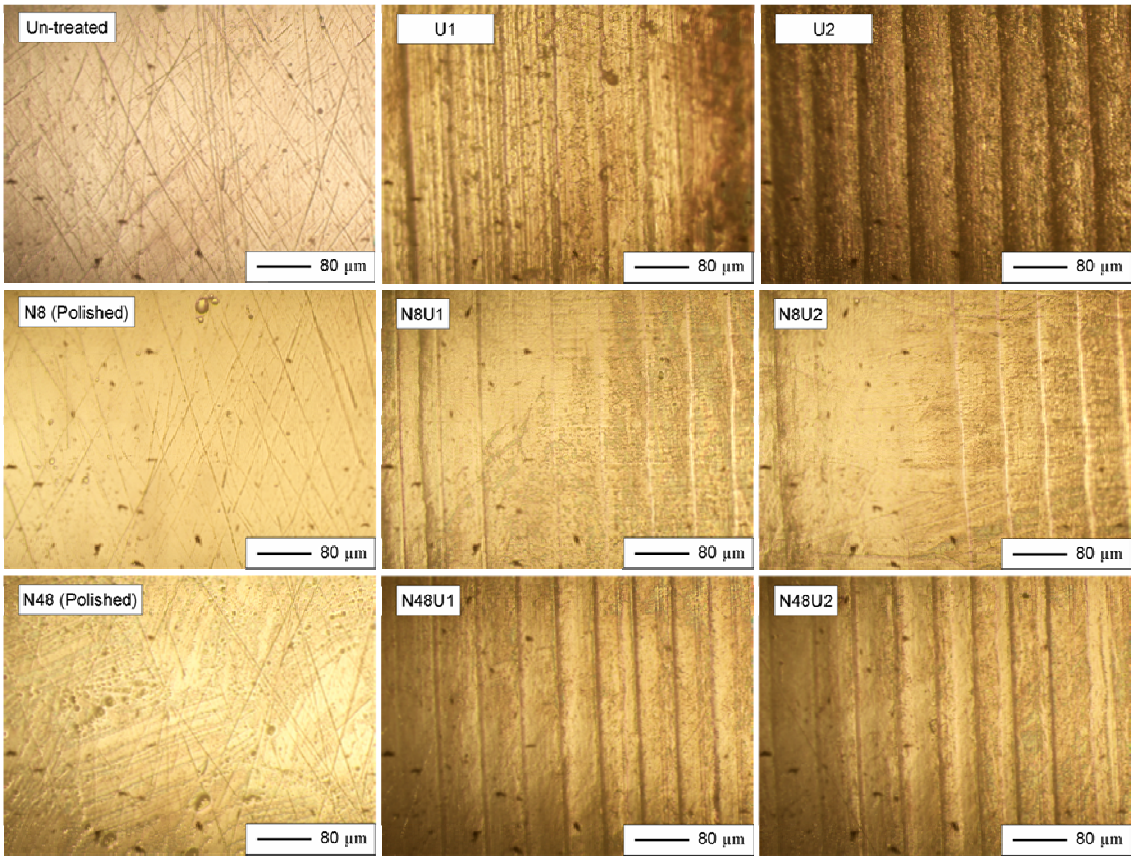

Figure 2. The surface morphology of specimens before and after UNSM.

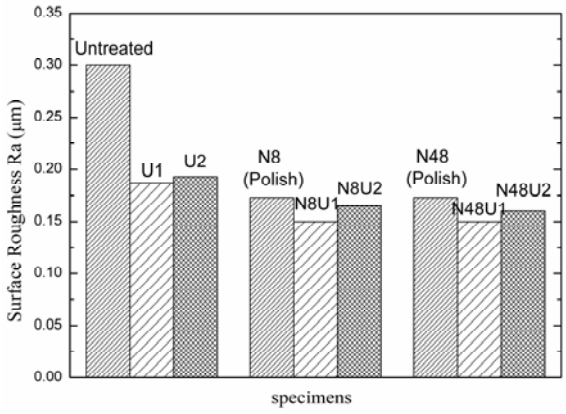

Figure 3. The surface roughness of specimens before and after UNSM.

The microstructures of the samples before and after UNSM treatment were shown in Figure 4. Through the other studies [3, 6] it can be seen that a gradual change microstructure from nano-sized grain in the sample top surface to the grain size of base material can be produced by the S^2PD treatment. With picric acid solution the grain boundaries can be etched easily. For the un-UNSMed samples the grain can be distinguished from top surface to interior and no difference between each

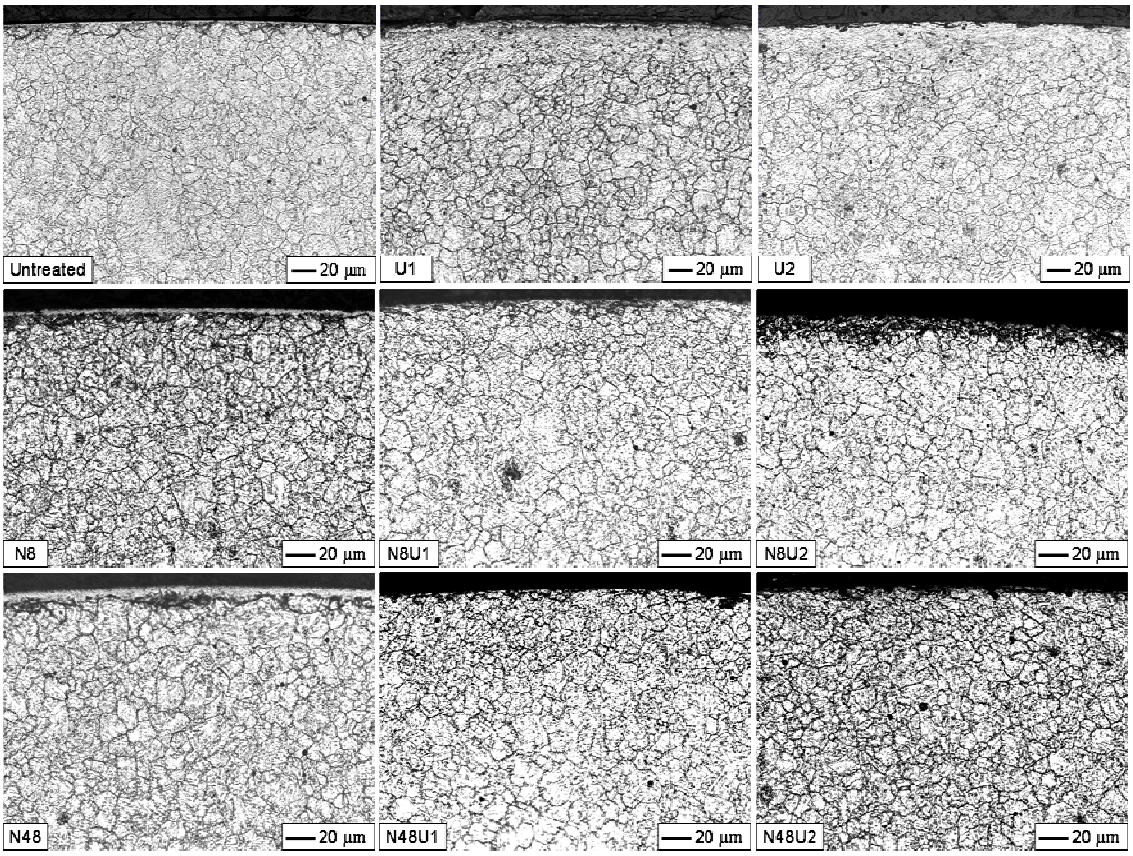

Figure 4. The microstructure of cross-section of specimens before and after UNSM.

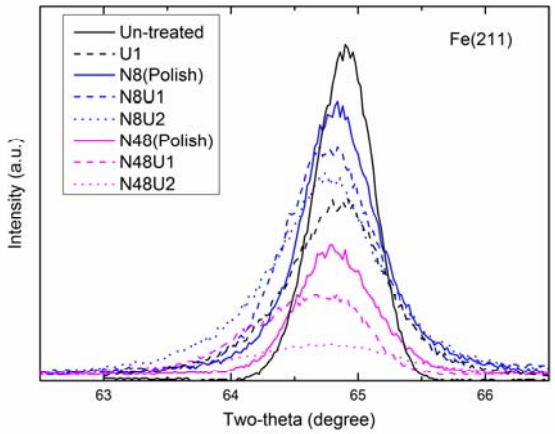

Figure 5. The XRD pattern of Fe (211) diffraction angles for the specimens before and after UNSM.

other can be observed. For the nitriding sample, a compound layer can be found on the nitriding sample surface (approximately 3 μm for N8 and approximately 8 μm for N48 sample). All the UNSMed samples can be found a grain refined layer at the edges of the cross-section. The refined layers are obvious for U1 and U2 samples approximately 40 μm for U1 specimens and approximately 70 μm for U2 specimens. However for the pre-nitriding samples, this layer is difficult to be distinguished and only some deformed grains can be found and the results show that the deformed layer induced by UNSM decreases with the increase of pre-sample surface hardness. The XRD was employed to scan the sample surface and the Fe (211) diffraction angles were scanned slowly and shown in Figure 5. Analyzing the XRD results and we can find that the Bragg diffraction peaks for all samples can be broadened by the UNSM. That means a grain refinement was induced on the sample surface according relation between grain size and the full width at half

maximum (FWHM). With FWHM results of all samples it can be found that though the nitrides within the sample surface zone and the XRD intensity of all un-UNSMed samples is different, the values of FWHM are close. And the degrees of the change are different. It is easy to find that the N48 samples were broadened easily compared with the un-treated and N8 samples. In many references [6-8], the hard phase can enhance the ability of the refinement of ferrite as different mechanism. So we can see that with the growth of nitrides concentration, harder sample surface can be obtained. More easy grain refinement can be found the harder sample, but thinner grain refined layer would be obtained as the reason of the surface with high hardness hindering the transfer of the plastic deformation energy produced by UNSM.

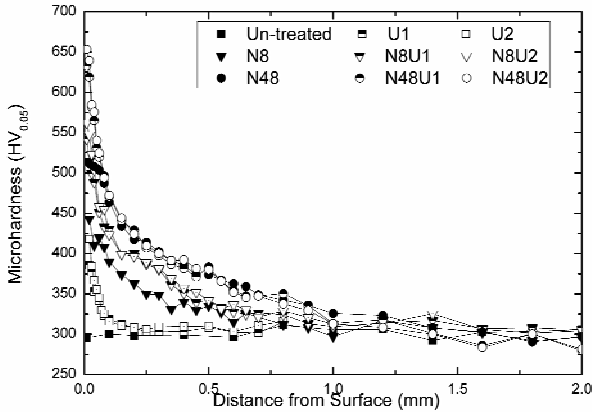

Figure 6. The microhardness of cross-section of specimens before and after UNSM.

The microhardness results of the all samples along the cross-section were shown in Figure 6. After UNSM treatment not only was surface hardness improved but also a gradual enhancement in the sub-surface region also can be observed. For the UNSM samples, the microhardness in the top nanostructured layer is 372 Hv for U1 sample and 418 Hv for U2 sample. For the N8 specimens, the surface hardness increased from 443 HV to approximately 540 HV after U1 treatment and to 560 HV after U2 treatment. These values were even higher than that of the N48 samples without UNSM treatment. For the N48 specimens, the surface hardness increased from 510 HV to 630 HV after U1 treatment and to 650 HV after U2 treatment. For all UNSMed samples the top surface hardness and the depths of effect increase with more strike number. It also can be found that the depths of effect regions are different with the change of samples. After the UNSM treatment, the materials surface was subjected to a severe plastic deformation induce a grain refinement. With the UNSM treatment, increased number of grain boundaries as the refined grains and generation of high density dislocation with strain hardening restrict the dislocation motion and render the material harder and stronger. According the microstructure results, we can also infer that the concentration of nitrides has a close connection with the hardening depth. Zhou [6] has found that compared with the strain-induced grain refinement in the pure Fe under the same SMAT processing, not only was refinement of ferrite much facilitated by the presence of dispersed cementite particles but also a thicker nanostructured layer can be obtained. The hard phase has the ability of transfer energy during S^2PD treatment. For soft surface materials, as the amplitude of 30 μm during UNSM processing energy of UNSM can not transfer too far as the absorption of energy by the top surface to have a plastic deformation. With the increase of nitrides concentration the hard phase of nitrides can transfer the energy to the deeper region. However when the surface hardness is too high, it is difficult to have a plastic deformation in the top surface and only induce a refinement of grains. So with just enough nitrides concentration, a deeper hardening layer can be produced.

The top surface residual stress was measured and shown in Figure 7. Before UNSM treatment un-treated and plasma nitriding samples all have a compressive residual stress on the surface

induced by QT (quenching and tempering) and nitriding process. After UNSM treatment the surface stress for all UNSMed samples was improved. However different trends with the increase of strike number can be observed for the UNSMed samples. For the un-treated sample, the surface residual stress increase from -217.4 MPa to -318 MPa with U1 and -395.1 MPa with U2 and it can be found that higher compressive residual stress can be obtained as the increase of UNSMed striking number. For the nitriding samples, the residual stress has a growth from -171.9 ~ -161.2 MPa (N8 and N48) to -925 MPa for N8 samples and -1089 MPa for N48 samples with U1 treatment. However a decrease (to -750 MPa for N8 samples and -778 MPa for N48 samples) was found with U2 treatment. This means that though with the growth of surface hardness higher compressive residual stress can be produced, a decrease was found as the increase striking number. This phenomenon is different from the U1, U2 results and other studies [2]. This may be the reason of the grain refinement of the nitrides in the surface zone inducing a relaxation of the surface stress.

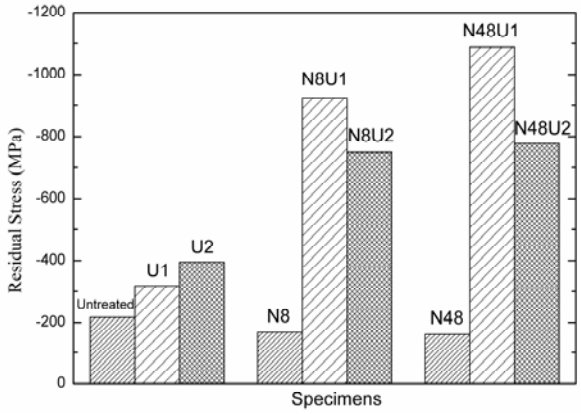

Figure 7. The residual stress on the surface of specimens before and after UNSM.

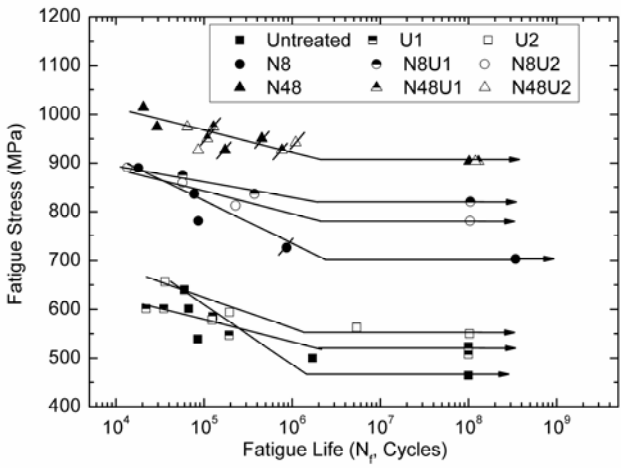

Figure 8. S-N curves of rotating-bending fatigue test

The fatigue test was employed on a rotating bending test machine and the S-N results are shown in Figure 8. The results show that after UNSM treatment the fatigue strength was enhanced for un-treated and N8 samples, but no improvement for the N48 samples. Compared with un-treated sample, more enhancements were obtained for N8 samples. At the same time the improvements of UNSMed samples have a close connection with the value of surface residual stress. Foe ever un-treated or N8 samples, the fatigue strength has a linear relation with the surface residual stress and with the higher residual stress, higher fatigue strength can be obtained. So we can find that a higher fatigue limit of N8 sample can be obtained after U1 treatment than after U2 treatment. It shows opposite compared with un-treated sample after UNSM treatment. This means that though

harder surface can be obtained by U2 treatment, the fatigue strength mainly depends on the level of surface stress. For the N48 samples, U1 process also introduced a higher compressive residual stress but the sub-surface fatigue crack initiation induced by inclusions hinders the improvement of fatigue strength.

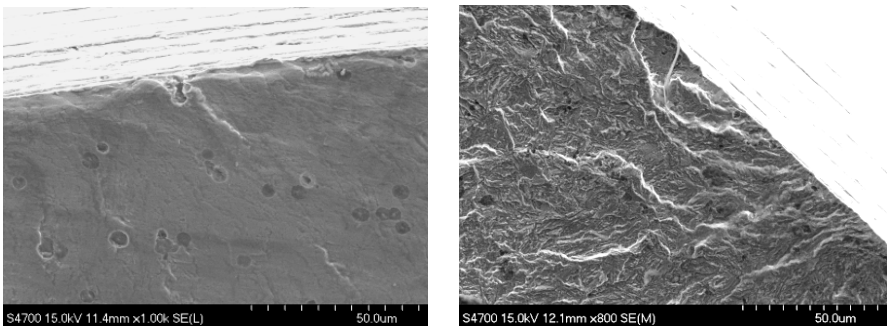

Figure 9. The fatigue crack initiating from surface defects (U2, 562MPa, $5.4x10^6$, left; N48U2, 974MPa, $6.46x10^4$, right)

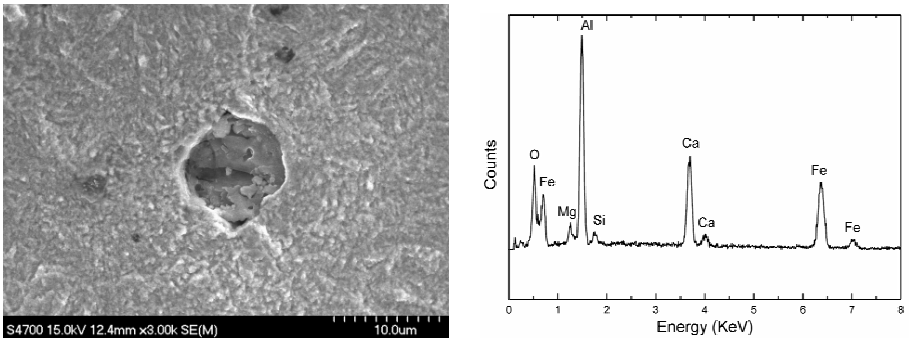

Figure 10. The sub-surface fatigue crack induced by inclusions (N48U1, 974.2MPa, $1.3x10^5$) (Left); EDX data on the inclusion.

The observations of the fatigue fractures were shown in Figure 9 and Figure 10. It obviously can be seen that the fatigue crack inducing the material's failure can be divided into two modes: one is the surface crack initiation induced by the persistent slip bands (PSBs) or surface defects; another is the sub-surface crack induced by the inclusions within materials. After plasma nitride, the surface of S45C steel can be enhanced effectively and the fatigue crack initiating from sample's surface becomes difficult. With the high cycle loading the inclusion within the sub-surface can induce a stress concentration and a fine granular area (FGA) (Figure 10, left), which was composed by the aluminum oxide, calcium oxide and magnesium oxide or other hard phases (Figure 10, right), was produced and expand to fracture. The mechanism of the formation of FGA is not clear and there are many theories explaining this phenomenon, for example, the theory of hydrogen diffusion, the dispersive decohesion of carbide and the grain refinement induced by stress concentration [9-11]. From the observations of fractures results, the UNSM treatment not only can improve the fatigue strength but also has an influence on the fatigue crack initiation. For the un-treated samples, the surface cracks transfer form surface crack induced by PSBs to the crack induced by the UNSM process marks (Figure 9, left) which we also can found in the surface morphology results. So we can also find that with increase of the test loading the fatigue life decreases as the reason of relaxation of residual stress and the stress concentration induced by the surface defects. For the nitriding samples, also were surface cracks induced by surface defects (Figure 9, right) found and the fatigue strength decreases with the increase of striking number (U2) as the reason of decrease of surface residual stress and more surface defects induced by the UNSM treatment after U2 process. We can also find more surface fatigue crack initiations for N48 samples with U1 treatment than U2

treatment. Combined with the results of surface roughness, it can be found that more surface defects can be introduced to the N48 samples' surface. So the fatigue strength is difficult to be enhanced as these defects inducing stress concentration easily. For the N48U1 samples, though high compressive residual stress and little defects were introduced, the inclusions within the sub-surface of samples still can form a fish-eye crack and hinder the improvement of fatigue strength. Also we found that as the high compressive residual stress induced by UNSM treatment the position of inclusions trend a deeper depth beneath the samples' surface [1]. With above results we can conclude that with the high loading during fatigue test the fatigue strength is more sensitive to the surface state of samples. At the same time, though high compress residual stress can be introduced by UNSM treatment, the sub-surface cracks induced by inclusions with materials are difficult to be hindered as the reason of limited depth of enhanced zone improved by UNSM treatment.

4. Conclusions

(1) With the increase of strike number, deeper UNSM processing marks can be introduced on the specimen surface; for the specimen with the higher surface hardness, little effect of UNSM to the surface roughness can be found.
(2) A gradual refined grain layer can be produced by the UNSM as multiple strikes to specimen surface; the thickness of the refined layer increases with the strike number and decreases as the growth of surface hardness; the top surface grains can be refined in to nano-size and for the sample with higher surface hardness grain refinement become easier.
(3) The surface hardness can be effectively improved by UNSM treatment and increases as the increasing strike number; N8 sample has a larger zone for the hardness increase as the effect of nitrides within sample.
(4) Higher compressive residual stress can be found for the nitriding specimens; increase of the residual stress can be obtained for un-nitriding samples as the increase of strike number; however opposite phenomenon was found for nitriding samples and residual stress decreases with the increasing strike number.
(5) The enhancement of fatigue strength mainly depends on the residual compressive stress in the surface zone produced by UNSM treatment; as the increase of fatigue strength the surface defects and sub-surface inclusions hinder the increase of fatigue strength definitely.

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