

Micro-Forging and Peening Aging Produced by Ultra-High-Temperature and Pressure Cavitation

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ABSTRACT

From microstructural examination of specimen surfaces or sides and from the mechanical properties resulting from compressive residual stress using Cr-Mo steel (SCM435), Ni-Cr-Mo steel (SNCM630), Ti-6aAl-4V, and Inconel (UNSN06601) processed by WJC and UTPC (micro-forging), the microstructure of the WJC-processed specimen revealed that voids and cracks tended to occur in the depth region of 0.5-1 mm from the topmost surface. The microstructure of the UTPC-processed specimen showed the spheroidization of cementite observed in the depth region of 0.5-1 mm from the topmost surface. In addition, voids and cracks were not observed in the specimen bulk. The Charpy impact energy of UTPC had the highest value of 101 J because ductile layers were formed by UTPC processed specimens for 5 hours. After stress relaxation testing, cracks owing to thermal stress do not occur at the grain boundary in the UTPC material having a tenacious tough layer inside. Micro-forging (UTPC) is promising for high-temperature oxidation of low-alloy steel, Ti alloy, and Inconel. Moreover, low-temperature and low-pressure cavitation (LTPC) is applied to age hardening of aluminum alloy Al-Mg-Si (AC4CH).

Keywords: Multifunction cavitation; Water jet cavitation; Ultrasonic cavitation; High-pressure high-temperature cavitation; Micro-forging; Peening aging

INTRODUCTION

Water Jet Cavitation (WJC) involves imparting mechanical energy to the surface of a material as the result of the high pressures generated during the collapse of a microjet close to the surface. Surface modification techniques such as WJC have been applied to reduce stress corrosion cracking in alloy steels and increase their fatigue strength. Surface modification technologies such as WJC have thus been applied to improve the stress corrosion cracking [1] and fatigue strength [2] of alloy steels. In the WJC process, high pressure [3] occurs when cavitation caused by high-pressure water jetted from a nozzle collapses on the metal surface. This impact pressure [4] results in slight plastic deformation of the surface layer and generates compressive residual stress by an elastic restraining force from the lower layer portion and the surroundings. When the compression force is converted to compression deformation, the deformation returns to its original state after the cavitation collapse; however, if a small amount of plastic deformation occurs, the compressive residual stress is generated after cavitation collapse. Another cavitation technique called Ultrasonic

Cavitation (UC) generates hot [5] spots that can promote chemical reactions by producing temperatures of several thousand degrees Kelvin [6]. Chemical and biomedical applications such as induced decomposition by the chemical action of UC have been investigated. We have previously reported Multifunction Cavitation (MFC) [7,8], which is a novel technique combining UC and WJC. In addition, we developed a new nozzle in order to greatly increase the MFC temperature and pressure, so allowing Ultra-High-Temperature And Pressure Cavitation (UTPC) to take place [9], which produces a new metal surface.

Low-alloy steel is based on carbon steel, but with a low percentage of alloying elements, in many cases less than 1%. Although its price is slightly higher than that of carbon steel, it offers improved resistance to specific types of corrosion. However, its corrosion resistance is still less than that of stainless steel. In conventional heat treatment processes for metals, heat treatment after cold working produces quite different results to cold working after heat treatment. In contrast, MFC has the capacity to allow microscale or nanoscale forging, in which the material is simultaneously worked and heat treated.

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Received: February 27, 2019; Accepted: March 12, 2019; Published: March 19, 2019

Citation: Yoshimura T, Ijiri M, Shimonishi D, Tanaka K (2018) Micro-Forging and Peening Aging Produced by Ultra-High-Temperature and Pressure Cavitation. Int J Adv Technol 9:227. doi: 10.24105/0976-4860.10.227

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The Ultra-High-Temperature And Pressure Cavitation (UTPC) took place in an aluminum alloy with a low melting point. The aluminum alloy used in this experiment was Al-0.31Mg-6.8Si alloy. Reinforcement of aluminum alloy is brought about because the supersaturated solid solution of elements added to aluminum precipitates at room temperature and becomes an obstacle to the movement of a dislocation. This is explained as an Orowan mechanism. This precipitation treatment is said to be aging, of which there are natural aging precipitated at normal temperature and artificial aging which is treated at a temperature of 200°C or less. In the case of T6 aging treatment, the Al alloy was kept at the temperature of about 150°C for several hours after solution treatment.

A precipitate called a GP zone precipitated from a supersaturated solid solution is considered to be Mg_2Si in the case of an Al-Mg-Si alloy. In any case, lattice distortion occurs due to precipitation of the GP zone, movement of the dislocation is inhibited, and a large force is required for deformation and strengthening. As the aging process progresses, it becomes a GP II phase or θ' phase, which are larger precipitates than GP I phase. However, when the stable I phase is reached, over-aging is reversed, and the Orowan reinforcement is reduced.

In the present study, UTPC has applied to the modification of Ni-Cr-Mo steel (SNCM630) surfaces, and the dependence of the microstructure and hardness on UTPC was investigated and the results compared to those for WJC. Furthermore, the toughness was evaluated by macroscopic mechanical strength (Charpy impact test) at room temperature, and the stress relaxation behavior and the crack resistance at a temperature of 500°C were investigated. Furthermore, the age hardening of the aluminum alloy (AC4CH) was studied by using low-temperature and Low-Pressure Cavitation (LTPC) in order to develop the technology of peening aging, which can impart high hardness and higher residual compressive stress simultaneously on the surface of the aluminum alloy.

EXPERIMENTAL

The materials used in this study were Cr-Mo steel (SCM435) and Ni-Cr-Mo steel (SNCM630), structural machine steel, and the chemical compositions of which are shown in Tables 1 and 2, respectively. In the case of the Cr-Mo steel, round bar (rod) specimens were heated at 870°C as a solution treatment, followed by quenching. Tempering was performed at 580°C. In the case of Ni-Cr-Mo steel,

Table 1: Charpy impact test results of Cr-Mo steel (SCM435).
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Test Pieces	Energy (J)*			
UTPC	101			
WJC	99			
As received	97			
Abrasive	96			
'Average value of three test pieces				

round bar (rod) specimens were heated at 860°C as a solution treatment, followed by quenching. Tempering was performed at 600°C. The specimens were subsequently cut into rectangular specimens with dimensions of $100 \times 100 \times 3 \text{ mm}^3$. In this study, we installed a straight swirl flow nozzle (inflow hole: 1 piece) [9] as shown in Figure 1 in the conventional nozzle, and bubbles were enlarged by low-pressure swirling flow and high-pressure cavitation was attempted. When high-pressure water is injected from the nozzle, the dynamic pressure at the nozzle outlet portion becomes very large, and conversely, the static pressure decreases so that the surrounding water flows in from the inflow hole of the swirl nozzle of Figure 1. As a result, a swirling flow (circulation) is generated in the swirl nozzle, and the circulation center is lowered. As a result of that, the bubbles expand, and if the expanded water jet bubbles are irradiated with ultrasonic waves, bubbles of higher temperature and pressure are generated than the conventional MFC, and the surface is micro-forged by UTPC. When two inflow holes are provided as shown in Figure 2, the inflowing surrounding water increases, the circulation center is further lowered, and it is possible to increase the size of the bubbles. Further, when the turning nozzle is tapered, the circulation radius gradually increases and the pressure at the



Figure 1: Generation of high-temperature and high-pressure cavitation by a straight swirling flow nozzle (Inflow hole: 1 piece).



Figure 2: Generation of high-temperature and high-pressure cavitation by a tapered swirling flow nozzle (Inflow hole: 2 pieces).

Table 2: Corrosion weight losses of Ti-6Al-4V alloy and Inconel material (Ti: sinking method, Inconel: coating method).

Specimen	Processing	Weight before corrosion (g)	Weight after corrosion (g)	Weight change (g)	Corrosion Weight loss (kg/m ²)
Ti-6A1-4V	As received	15.9821	15.9748	-0.0073	0.00304
Ti-6A1-4V	SFN-WJP	15.9791	15.9768	-0.0023	0.00096
Ti-6A1-4V	SFN-MFC	15.8784	15.8788	0.0004	-0.00017
Inconel (UNSN06601)	As received	22.1889	22.1815	-0.0074	0.00308
Inconel (UNSN06601)	SFN-WJP	22.1762	22.1754	-0.0008	0.00033
Inconel (UNSN06601)	SFN-MFC	22.2572	22.2563	-0.0009	0.00038

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circulation center decreases so that it is possible to further increase the size of the bubble and achieve high temperature and high pressure.

In a conventional WJC apparatus, a nozzle is fixed in water at room temperature and water jets are pumped at a discharge pressure of 35 MPa. The nozzle diameter used in the present work was 0.8 mm. The distance between the nozzle and the specimen was set to be 65 mm. In UTPC treatment, an ultrasonic transducer is set to the vertical direction of the water jet nozzle for conventional WJC, and ultrasonic waves are sonicated to the water jet flow from the water jet nozzle. During the present study, UC was performed at 800 W and 28 kHz. Residual stress measurement, hardness testing, optical microscope observation, and Scanning Electron Microscope (SEM) observation were performed under the same conditions. In preparation for observations, specimens were corroded in 5 vol% Nital etchant solutions. Prior to all post-processing characterization procedures, specimens were cut into 1 × 1 cm² test samples.

The Charpy impact tests were carried out in order to investigate the improvement of brittleness by peening. Furthermore, stress relaxation tests were carried out by heating the cavitation-processed specimens in an electric furnace at a temperature of 500°C. The stress relaxation behavior of UTPC specimens was compared to that of WJC specimens.

A test piece subjected to various cavitation treatments was inserted into a crucible, and the crucible was inserted into an electric furnace and removed. It was descaled with a solution heated with a ceramic heater and the weight change was measured. The chemicals simulating the corrosive environment were sodium sulfate (Na₂SO₄) and potassium sulfate (K_2SO_4), the application was performed on only one side of the test piece, and the above chemicals were dissolved in water and applied on the construction surface. In some cases, a sinking method was adopted in which chemicals mixed at a weight ratio of 1:1 was placed in a crucible and the specimen (double-sided treatment) was subjected to a hightemperature furnace of 500°C. In some cases, the specimen (singlesided treatment, chemical coating method) was directly subjected to a high-temperature furnace of 500°C.

In order to develop the technology of peening aging, LTPC of the aluminum alloy Al-0.31Mg-6.8Si was processed by a tapered swirling nozzle (inflow hole: 2 pieces) (Figure 2). The water jet pressure and the ultrasonic output were optimized for higher hardness and higher residual compressive stress imparted on the surface of the aluminum alloy. The discharge pressure of the water jet decreases from 35 MPa to 20 MPa, which leads to a low collapsed pressure of microjets. Furthermore, the output of the ultrasonic wave decreases from a maximum of 800 W to 100 W, which provides a low collapsed temperature of microjets.



Figure 3: Mechanisms of micro-forging by microjets of UTPC and peening aging by microjets of LTPC.

Mechanisms of micro-forging by microjets of UTPC and peening aging by microjets of LTPC (Figure 3). In the case of micro-forgingby UTPC, the high impact pressure of microjets generated a lot of dislocations owing to work hardening, and large precipitations like the spherical cementite in low-alloy steel and the Mg-Si-Fe rich precipitations in the aluminum alloy as described later are formed because of high surface temperature. After the disappearance of cavitation, compressive residual stress and an increase of hardness are imparted by elastic constraint from the surroundings. On the other hand, in the case of peening aging by LTPC, the number of dislocations is not higher than UTPC because of the low impact pressure of microjets. However, a lot of Mg-Si rich precipitations (GP zone) are formed and the entanglement of dislocations and precipitations occurs, which leads to the increases of compressive residual stress and hardness.

EXPERIMENTAL RESULTS AND DISCUSSION

Micro-forging by UTPC

From SEM observation of a specimen side of Cr-Mo steel (SCM435) after WJC processing for 2 min, overall, the lamellae layer of pearlite had a collapsed shape and the ferrite 10 min. No grains were elongated. It was confirmed that cracks occurred around the ferrite grains at a depth of approximately 0.5-1 mm from the surface [10]. Voids or cracks occurred mainly at grain boundaries in other places where ferrite and pearlite grains were in contact. Similar features were also observed after WJC processing for voids or cracks were observed at depths of approximately 2-3 mm from the surface. Thus, WJC processing imparts compressive residual stress without changing the properties of the material surface. However, it was revealed that voids and cracks are likely to be formed at grain boundaries where ferrite and pearlite grains are in contact within the specimen bulk after WJC processing [11]. The specimen surface after UTPC processing for 2 min was observed using SEM; however, ferrite and pearlite similar to the specimen surface after WJC processing were not observed [10]. In the cross-sectional SEM images of the specimen side in the vicinity of the surface after MFC processing of Cr-Mo steel, decoupling of cementite in pearlite was recognized. Furthermore, the formation of spheroidized structures observed by heat treatment of cementite was observed. This also suggests a high surface temperature at the time of UTPC processing [10].

There are two types of fracture surface, which are ductile and brittle. The ductile fracture surface has a dimpled structure, whereas the brittle fracture surface has flat areas due to grain boundary fractures. In the present study, the MFC processing was carried out on the four faces of a Charpy impact test specimen with a cross-sectional area of $10 \times 10 \text{ mm}^2$. Table 1 shows the results of Charpy impact testing. There are no remarkable differences among the Charpy impact energies of test pieces. However, the impact energy of UTPC has the highest value at 101 J. This is because the ductile layers were formed by UTPC processing. The proportion of ductile and brittle in UTPC processing is higher than the other specimens as shown in Figure 4.

Stress relaxation behavior of various processed materials at a temperature of 500°C. In order to increase the residual compressive stress, initially, the surface is ground in order to provide the tensile residual stress. The surface tensile stress gives higher compressive stress after peening because of the shakedown effect. Figure 5 shows the residual compressive stress along the vertical grinding



Figure 5: Stress relaxation behavior of various processed materials (Cr-Mo steel (SCM435), 500°C, and vertical grinding direction).

direction. Compressive residual stress of more than 100 MPa is retained after annealing both the WJC- and UTPC-processed specimens for 5 hours.

The results of observing the cross-section of the same test piece are shown in Figure 6. As shown in the figure, very long cracks are generated in the grain boundaries of ferrite-pearlite in the crosssection immediately below the surface of the untreated material. Cracks were also observed in the WJC material between ferrite and pearlite, for example.

This is thought to be caused by thermal stress accompanying the temperature distribution on the surface and inside. In particular, it is considered that cracks are likely to occur between the hard pearlite layer and the soft ferrite layer because there is no tough layer just under the surface of the WJC-treated material. Voids and cracks do not occur between ferrite and pearlite, even if the UTPC material of Cr-Mo steel (SCM435) is held at 500°C for 5 hours. This indicates that the toughness of the micro-forged material is retained. As described above, the surface crack of the untreated material disappears in the micro-forged material.

On the other hand, it was treated by micro-forging (super-hot temperature high-pressure cavitation, the straight swirling nozzle (Figure 1), ultrasonic output: 800 W, ultrasonic mode: Dual, construction time: 2 min). Figure 7 shows the relationship between corrosion number and corrosion weight loss. UTPC processing (MFC) is most effective at suppressing corrosion weight loss compared to the other specimens (WJP and as received).

Table 2 shows a corrosion weight loss of Ti-6Al-4V alloy and an Inconel material (Ti: sinking method, Inconel: coating method). For the Ti-6Al-4V alloy, the as-received material has a corrosion weight loss of 0.00304 kg/m², whereas the micro-forged material is not corroded at all. This shows that micro-forging is promising for high-temperature oxidation. Further, even with the Inconel material, the corrosion weight loss of the untreated material is 0.00308 kg/



(a) As received

(b) WJC processing



(c) UTPC processing

Figure 6: SEM microstructure photographs of cross-sections (a) as received and (b), (c) after stress relaxation test (SCM435, $500 \,^{\circ}\text{C} \times 5 \,\text{h}$).



Figure 7: Relationship between corrosion number and corrosion weight loss.

 m^2 , whereas that of the micro-forged material is 0.00038 kg/m², which is a reduction to 1/10 or less. There is a great expectation for future improvement of high-temperature corrosion resistance of Ti-6Al-4V alloy and Inconel material.

The micro-forging in this study not only imparts a high compressive residual stress on the surface but also improves corrosion resistance and has the feature of forming a tough layer just under the outermost surface. Even if a stress relaxation test at 500°C is performed on this characteristic surface, the crack resistance performance is very high and cracking does not occur. By optimizing the processing conditions of micro-forging, it was shown that it is promising for high-temperature oxidation resistance.

Peening aging by LTPC

The processing by ultra-high-temperature and pressure cavitation is referred to as peening aging treatment because this has a peening effect of imparting residual stress, work hardening, and precipitation strength. The mechanism of peening aging treatment is shown in Figure 8. When Ultra-high-Temperature and Pressure Cavitation



Figure 8: Mechanisms of peening aging by UTPC and LTPC processing.

(UTPC) collapses on the material surface, a high-temperature and high-pressure microjet plastically processes the surface and causes many dislocations on the surface. At the same time, it undergoes aging treatment accompanying temperature rise and precipitation hardens. Dual 800 W, which has the highest ultrasonic power output at a high water jet pressure of 35 MPa, which in turn is the condition for producing spherical cementite in low-alloy steel, as described above, has a high temperature, so the age hardening is small and work hardening becomes rate-determining due to the high pressure.

On the other hand, under conditions of LTPC (Low-Temperature and low-Pressure Cavitation) where the water jet pressure is low and ultrasonic output is low, age hardening is dominant because of the low temperature, and occurrence of dislocation due to work hardening is suppressed due to the low pressure. Furthermore, in the test piece subjected to the T6 aging treatment, Mg,Si is already precipitated by aging but there is no compressive residual stress on the surface. When subjected to UTPC treatment, work hardening by microjet is applied to impart compressive residual stress. However, work hardening is rate-limiting due to a high pressure of 35 MPa, because aging is hard to rise because of high-temperature microjets under the condition of ultrasonic output, 800 W. On the other hand, under conditions of low water jet pressure, 20 MPa, and ultrasonic output, 100 W, because of the low temperature, age hardening progresses further and precipitation strengthening occurs, so dislocation generation due to work hardening is suppressed because of the low pressure. However, when aging progresses excessively, a stable θ phase precipitates and becomes excessive aging and attention to softening is required.

The relationship between processing time and hardness is shown in Figure 9. Hardness increased rapidly in 2 min, and it gradually increased to 30 min thereafter. If MFC processing with a straight swirling nozzle is additionally applied to the T6-treated material, it hardens rapidly in 2 min like other cavitation materials, but in 30 min it has the same hardness as the MFC-processed materials without T6 treatment. This indicates that the sum of work hardening and age hardening has reached the same level as explained in Figure 8.

When concentration fluctuation occurs, it is considered that the Mg concentration distribution becomes broad and the standard deviation σ increases as shown in Figure 10. In addition, Mg was assumed to diffuse into the grain from the grain boundary, and it was assumed that a high concentration shift occurred in which the 6

The results analyzed by the X-ray crystal structure analysis (XRD) apparatus revealed the peak at $2\theta=46^\circ$ corresponding to Mg₂ Si in the case of SFN-MFC, the same as T6 aged material. These peaks suggest the formation of GP zones I and II, and that intermediate phase (θ) and stable phase (θ) formed within the grains. On the other hand, the peak at $2\theta=46^{\circ}$ was not recognized in the untreated specimen.

material.

Element mapping of the Al-Mg-Si alloy surface by LTPC processing and T6 aging is shown in Figure 11. The grain part of the UTPC specimen with a high concentration of Mg is found to be the same as that after T6 aging. However, in the case of LTPC, the Mg-Si-Fe rich precipitations were locally observed besides the above grain parts with a high concentration of Mg. Here, the conditions of LTPC were water jet pressure of 20 MPa, the ultrasonic output of



Figure 9: Relationship between processing time and Vickers hardness (Water jet pressure: 35 MPa).



Figure 10: Distribution of Mg concentration and high concentration shift in precipitation aging and spinodal decomposition by LTPC treatment.



Figure 11: Element mapping of Al-Mg-Si alloy surface by LTPC (20 MPa, 100 W, 20 min) processing and T6 aging.

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100 W, working time of 20 min, and the addition of a tapered swirling nozzle with an inflow hole of 2 pieces. The number of MgSi-Fe rich precipitation tends to increase with pressure and temperature of cavitation bubbles.

Figure 12 shows profiles of Mg concentration of an as-received specimen and of a specimen after LTPC treatment. Here, these profiles were measured in each crystal grain by SEM-EDS analysis. It should be noted that the high concentration shift in precipitation aging of the specimen after LTPC treatment was recognized. From these profiles of Mg concentration, distributions of Mg concentration with average value and broadness are obtained.

Figure 13 shows distributions of Mg concentration obtained by line analysis of low vacuum SEM in crystal grains on a specimen side of distance 10 μ m to 40 μ m from the topmost surface. It should be noted that the Mg concentrations after T6 aging, MFC 30 MPa 200 W and MFC 35 MPa 800 W are shifted from the as-received concentration. The highest shift was the concentration of MFC 30 MPa 200 W. These results were in accordance with those of Figure 10.

Figure 14 is the relationship between ultrasonic output (sound pressure in water) and compressive residual stress. The highest residual stress was obtained at the water jet pressure of 100 W.



Figure 12: Profiles of Mg concentration of as-received specimen and specimen after LTPC (20 MPa, 100 W, 20 min) treatment.



Figure 13: Distribution of Mg concentration obtained by line analysis of low vacuum SEM in crystal grains on specimen side of distance 10 μ m to 40 μ m from the topmost surface (Tapered swirling nozzle, Inflow hole: 2 pieces).



Figure 14: Relationship between ultrasonic output (sound pressure in water) and compressive residual stress (20 MPa, 20 min).



Figure 15: Relationship between water jet pressure and compressive residual stress (100 W, 20 min).

The residual compressive stress imparted on the surface shows a peeling effect. As described before, the aging of aluminum alloy is thought to progress at a lower temperature than that of low-alloy steel. Therefore, LTPC is more appropriate than UTPC, in the case of age treatment of aluminum alloy.

The relationship between water jet pressure and compressive residual stress is shown in Figure 15. It was found that the compressive residual pressure decreases with the progress of water jet pressure increase. The highest residual stress was obtained at the water jet pressure of 20 MPa. As indicated in Figure 8, the age hardening becomes larger than the work hardening and an increase of residual stress was provided by elastic constraint. It is known that too large a shear stress generates a dislocation loop after dislocations going through precipitates. Therefore, the compressive stress decreases by the elastic constraint reversely decreasing, because the Orowan mechanism does not become effective. It is thought that when the surface deformation force by microjets due to a water jet pressure of 20 MPa is smaller than that of 35 MPa and the dislocations depending on precipitates are more, and more proper than those of 35 MPa, this leads to the higher compressive residual stress produced by elastic constraint from around the peening area.

CONCLUSION

From a microstructural examination of specimen surfaces or sides and from the mechanical properties resulting from compressive residual stress using Cr-Mo steel (SCM435), Ni-Cr-Mo steel (SNCM630), Ti-6aAl-4V, and Inconel (UNSN06601) processed by WJC and UTPC, the following conclusions were obtained:

(1) The microstructure of the WJC-processed specimen revealed

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that voids and cracks tended to occur in the depth region of 0.5-1 mm from the topmost surface. Moreover, voids and cracks were not observed at depths of 2-3 mm from the surface

(2) The microstructure of the UTPC-processed specimen showed the spheroidization of cementite observed in the depth region of 0.5-1 mm from the topmost surface. In addition, voids and cracks were not observed in the specimen bulk

(3) The Charpy impact energy of UTPC had the highest value of 101 J because the ductile layers were formed by UTPC processing. The proportion of ductile and brittle in UTPC processing is higher than the other specimens

(4) Stress relaxation behavior of various processed materials at a temperature of 500°C was investigated. Compressive residual stress of more than 100 MPa was retained after annealing both the WJC and UTPC processed specimens for 5 hours

(5) After stress relaxation testing, cracks owing to thermal stress do not occur at the grain boundary in the UTPC material having a tenacious tough layer inside

(6) Micro-forging (UTPC) is promising for high-temperature oxidation of low-alloy steel, Ti alloy, and Inconel

(7) LTPC (Low-Temperature and low-Pressure Cavitation) is effective for the age hardening of aluminum alloys

ACKNOWLEDGMENT

This work was supported by the Innovative Science and Technology Initiative for Security program of the Acquisition, Technology and Logistics Agency (ATLA) of Japan. This research was also supported in part by JSPS KAKENHI Grant Number 16K06029 and the Light Metal Educational Foundation, Inc.

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