

Using Sub-second Electric Pulsing, a Microstructure Reset-Based Self-Healing Method for Metallic Materials

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Abstract

Microscale defects in materials damage the originally designed microstructure during their use, degrading their mechanical properties/life expectancy. Therefore, various methods for damage repair in materials have been proposed based on the concept of self-healing. However, self-healing for bulk metallic materials is still a challenge due to their strong atomic bonding. Here, we introduce a sub-second electric pulsing which can enhance the kinetics of microstructural changes to infinitely reset the damaged microstructure of metallic materials as a non-autonomous self-healing method. The principle of microstructure resetting is explained based on three categories of resetting cores: phase transformation, dislocation recovery, and recrystallization. Microstructure resetting assisted infinite reuse is successfully realized using 301L/316L stainless steels and super-elastic NiTi alloy, which are applicable materials of the resetting core [1-15]. This is a new concept combining extreme simplicity, rapidness, and infinite repetition, which cannot be achieved by conventional methods. Metallic materials are the most widely used in all engineered structures. They inevitably accumulate microscale damages such as second phase, dislocation, and micro voids during their use. These damage the originally designed microstructure, which degrades the mechanical properties/life expectancy of the engineered structures, and even leads to failure. Therefore, various methods for healing or repairing the damages in materials have been proposed based on the concept of self-healing

Introduction

In the early stage of self-healing, polymer materials were focused due to their intrinsic ability to achieve autonomous healing of damage However, applying self-healing to metallic materials is intrinsically difficult due to their low atomic mobility or diffusivity, their high melting points, and the difficulty encountered in detecting damage . In order to apply the self-healing concept to metallic materials, many researchers have adopted two types of method in a methodological point of view autonomous self-healing of defects at the nanoscale, aiming at a prevention of large-scale damage, and nonautonomous self-healing of microcracks by applying external stimuli such as heat, magnetic field, and pressure to repair the damaged microstructure.

First, the autonomous self-healing in metallic materials was proposed with the adoption of precipitation formation at high. For this case, the original microstructure must contain a supersaturated amount of solute atoms. When defects form at a grain boundary during damage, the solute atoms move to the defect which acts as a nucleation site for the precipitation process so that the defect can be healed through the precipitation formation. However, this concept requires high temperature and much time for healing. In addition, since a high temperature atmosphere must be continuously maintained, it is limitedly applied for the purpose of improving creep resistance.

Subjective Heading

Naturally, the non-autonomous self-healing concept using various stimuli including thermo mechanical process was preferred for metallic materials. One approach was using solder capsule/wire with low melting temperature inside metal]. The solder is activated only when a crack has formed in the matrix. If the temperature is increased above the melting temperature of the solder, the melted solder fills the crack. However, since the capsule/wire need to contain hole so that the solder should be filled in, designing the original microstructure is difficult. Also, this requires an enhanced interfacial bonding be

Microscale flaws in materials degrade mechanical characteristics

and life expectancy by causing damage to the initially specified microstructure during usage. As a result, based on the concept of self-healing, numerous approaches for material damage repair have been presented. Due to their strong atomic bonding, self-healing for bulk metallic materials remains a difficulty. As a non-autonomous self-healing approach, we introduce a sub-second electric pulsing that can improve the kinetics of microstructural changes to infinitely reset the damaged microstructure of metallic materials. Three types of resetting cores are used to explain the principle of microstructure resetting: phase transformation, dislocation recovery, and recrystallization. 301L/316L stainless steels and super-elastic NiTi alloy, which are appropriate materials for the resetting core, have been successfully used to achieve microstructure resetting assisted infinite reuse. This is a novel notion that combines extreme simplicity, rapidity, and endless repetition in a way that conventional methods cannot.

Discussion

Metals are the most commonly utilized materials in all engineered buildings. During their use, they will inevitably collect microscale defects such as second phase, dislocation, and microvoids. These harm the microstructure that was initially created, reducing the mechanical qualities and life expectancy of the manufactured structures, and even causing failure. As a result, based on the concept of self-healing,

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different ways for healing or correcting material damages have been proposed.

First, with the use of precipitation production at high temperatures, autonomous self-healing in metallic materials was proposed the initial microstructure must have a supersaturated amount of solute atoms in this situation. When defects form at a grain boundary as a result of damage, the solute atoms travel to the defect, which functions as a nucleation site for the precipitation process, allowing the defect to be healed. However, this notion necessitates a high temperature and a long healing time. Furthermore, because a high temperature environment must be maintained continually, it is only used to improve creep resistance.

Therefore, the method using only conventional heat treatment (HT) has been proposed without new material design for the nonautonomous self-healing in metallic materials the heat of high temperature enhances the atom mobility, which tends to drive the system towards a reduction of the excess surface energy. If the preferred structure of the material is well defined, the damaged structure may be repaired by the increase in temperature. However, to heal sizeable cracks, the temperature needs to be raised significantly and sufficient time should be needed. To overcome above drawback, it was proposed that an effective healing of internal cracks in the matrix can be achieved using the combined effect of high temperature and compressive stress It was demonstrated that self-healing of internal cracks could be achieved in a low-carbon steel when hot plastic deformation was applied at temperatures ranging from 900 to 1200 ° However, this is far from the self-healing concept and is unpractical in that it requires additional deformation at high temperature.

In order to overcome these weak points of already proposed nonautonomous self-healing processes for metallic materials, we propose a sub-second electric pulsing as a new non-autonomous self-healing concept to effectively and infinitely reset the damaged microstructure of metallic materials to its original microstructure. When a highdensity electric pulsing is applied to metallic materials during deformation, the ductility is significantly improved with reduced flow stress, which is called "electro plasticity". Until now, research on electro plasticity has received remarkably increasing attention in both academic and manufacturing fields. Many researchers have examined the effect of electric current on the mechanical behavior of various metallic materials based on microstructural observations For example, the annihilation of dislocation was accelerated by applying pulsed electric current during tensile test of aluminum and titanium alloys . The formation of early precipitation or atomic clustering from a supersaturated state, i.e., aging process, was enhanced by applying pulsed electric current during the tensile test of 6061 aluminum alloy Moreover, electro pulsing treatment was proved to accelerate recrystallization kinetics when compared to that of the furnace HT for steel These phenomena show an obvious a thermal effect caused by electric current, which is distinct from the thermal effect caused by temperature rise due to Joule heating. Thus, it can be said that electric current can enhance the kinetics of microstructural changes due to the thermal effect. In addition, electric pulsing provides benefit such as rapid heating up to a target temperature in only 0.01-1.0 s

In this study, based on the positive effects of electric pulsing, we design a strategy of microstructure resetting for infinite reuse of metallic materials. For the selection of materials, resetting core which reflects a reversible damage characteristic will be defined and classified into three categories: phase transformation, dislocation recovery, and recrystallization. As representative materials to which the resetting cores can be applied, 301L/316L stainless steels (SUS301L/SUS316L) and super-elastic Nit alloy are selected. To prove the microstructure resetting assisted infinite reuse, we repeatedly conduct the deformation and electric pulsing treatment. The principle of microstructure reset-based self-healing method will be discussed from various microstructural observations with a focus on the resetting core.

Three materials having resetting cores, commercial 301L/316L stainless steels (SUS301L/SUS316L) and super-elastic NiTi alloy were selected. The chemical compositions of the SUS301L and SUS316L are 0.03C-0.74Si-17.93Cr-0.59Mn-6.41Ni-Bal. Fe and 0.08C-0.53Si-16.80Cr-1.08Mn-10.10Ni-Bal. Fe by wt. %, respectively. SUS301L, which is one of the transformation-induced plasticity (TRIP) steels, consists of a metastable austenie phase. Generally, the metastable austenite transforms into a hard marten site phase (body centered cubic, b.c.c. structure) when the sum of mechanical energy due to the externally applied stress and the chemical driving force exceeds a critical value . In contrast, SUS316L is well known to have higher stability in the austenite phase than SUS301L, resulting in plastic deformation via dislocation evolution or These two materials are most widely used due to the superior mechanical properties such as ductility, corrosion resistance etc.

To allow the electric current to circulate in the sample, a custommade fixture was created. We were able to relieve stress by allowing the sample to move in both directions, which solved the problem of bending produced by rapid thermal expansion when the sample was subjected to electric pulsing. Using a FLIR-E40 infrared (IR) thermal imaging camera, we observed the temperature change of the sample during pulsing. To improve the accuracy of temperature measurement, one side of the sample was painted with black thermal paint to stabilize the emissivity. Using a K-type thermocouple, the emissivity was calibrated by comparing the observed temperature. When the electric pulse was applied to the sample, the temperature was measured at the center of the specimen, which was the part at maximum temperature.

To achieve the best results, we changed the electric current density and duration based on the chosen target temperature. According to the equipment specifications, the optimal electric current density for safely applying electric current to the SUS301L and SUS316L was 90 A/mm². For SUS301L and SUS316L, the ideal electric current durations were 0.55 and 0.65 seconds, respectively. The ideal electric current density and duration for the super-elastic alloy were 100 A/mm² and 0.30 s, respectively.

NiTi alloy (Nitinol) also consists of a metastable austenite phase. The chemical composition is 55.8Ni-0.05Fe-0.05C-0.025N-0.025O-Bal.Ti by wt.%. Under an external stress, the metastable austenite phase (B2 phase, b.c.c. structure) transforms to the martensite phase (B19' phase, monoclinic structure) Once the stress is removed, it returns to its original shape as the martensite phase fully transforms back to the austenite phase, which is called super-elasticity (SE) effect. This transformation is diffusionless and reversible stress-assisted martensitic transformation of a metastable austenite. This unique property and high biocompatibility make the super-elastic NiTi alloy a top choice material for biomedical applications or next-generation refrigeration systems.

Tensile samples of the SUS301L and SUS316L with a gauge width of 9 mm, thickness of 1 mm, and gauge length of 12 mm were fabricated along the rolling direction (RD) of the sheet. For the super-elastic NiTi alloy, the sample was fabricated along the transverse direction (TD) of the sheet by halving the sample dimension of the SUS301L and

SUS316L (Fig. S1a).

Instrumental set-up

The uniaxial tensile tests were conducted using a tensile test machine (INSTRON 5584, USA) at a constant crosshead speed of 1.5 mm/min at room temperature (RT, 25 °C). The cyclic deformation was performed using a tensile test machine (INSTRON 5582, USA) at a constant crosshead speed of 1.0 mm/min at RT. The sample displacement was measured using the ARAMIS Digital Image Correlation (DIC) system (GOM, Germany), which enabled non-contact measurement based on the principle of digital image correlation.

A custom-made fixture was designed to allow the electric current to flow in the sample. Specifically, we made it possible to relieve the stress by making the sample move in both directions to solve the problem of bending caused by the rapid thermal expansion when the electric pulsing was applied to the sample (Fig. S1b). The electric pulsing was generated by a Vadal SP-1000U welder and a Keysite 6680A power supply.We measured the temperature change of the sample during pulsing using an FLIR-E40 infra-red (IR) thermal imaging camera (FLIR, Sweden). One side of the sample was sprayed with a black thermal paint to stabilize the emissivity and thus improve the accuracy of temperature measurement. The emissivity was calibrated by comparing the measured temperature using a K-type thermocouple. The temperature was measured at the center of the specimen, which was the portion at maximum temperature when the electric pulsing was applied to the sample.

Conclusion

Figures S2a, b, and c show the temperature variations of the sample during pulsing treatment for the SUS301L, SUS316L, and super-elastic alloys, respectively. Due to Joule heating, the sample temperature increased rapidly after each pulsing treatment. The sample temperature was reduced by air cooling after applying electric pulse. During each pulsing treatment, the measured temperature peak values were extremely close to the target temperature. This is because, in order to maintain the electric current density constant, the amplitude of the electric current was altered in response to the decrease in cross sectional area of the sample caused by deformation.

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Conflict of Interest

The authors declare that they are no conflict of interest.

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