TITLE:
An available technique for preparation of new cast MnCuNiFeZnAl alloy with superior damping capacity and high service temperature

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Mn-Cu based damping alloys, Sand mold casting, Damping capacity, Service temperature, Heat treatment, Martensitic transformation

LONG ABSTRACT:
Manganese-copper based alloys have been found to have damping capacity and can be used to reduce harmful vibration and noise effectively. M2052 (Mn-20Cu-5Ni-2Fe, at. %) is an important branch of Mn-Cu based alloys, which possesses both excellent damping capacity and processability. In recent decades, lots of studies have been carried out on the performance optimization of M2052, including improving the damping capacity, mechanical properties, corrosion resistance and service temperature *etc*. The major methods of performance optimization are alloying, heat treatment, pretreatment, different ways of molding *etc*., among which alloying as well as adopting reasonable heat treatment is the simplest and the most effective method to obtain perfect and comprehensive performance. To obtain the M2052 alloy with excellent performance for casting molding, the research group proposes to add Zn and Al in the MnCuNiFe alloy matrix and uses a variety of heat treatment methods for comparison in microstructure, damping capacity and service temperature. Finally, a new type of cast-aged Mn-22.68Cu-1.89Ni-1.99Fe-1.70Zn-6.16Al (at. %) alloy with superior damping capacity and high service temperature was obtained by an optimized heat treatment method. Compared with forging technique, cast molding is simpler, more efficient and the damping capacity of this as-cast alloy is excellent. Therefore, there is a suitable reason to think that it is a good choice for engineering applications.

INTRODUCTION:
Since the manganese-copper alloys were found to have damping capacity by Zener[1], they have received widespread attention and research[2]. The advantages of Mn-Cu alloy are that it has high damping capacity, especially at low strain amplitudes, and its damping capacity cannot be disturbed by magnetic field, which is quite different from ferromagnetic damping alloy. The

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high damping capacity of Mn-Cu based alloys can be mainly attributed to the movability of internal boundaries, mainly including {110} twin boundaries and phase boundaries which generated in the face centered cubic to face centered tetragonal (f.c.c.-f.c.t.) phase transition under the martensite transformation temperature (T_t)[3]. It has been found that T_t depends directly on the Mn content in the Mn-Cu based alloy[4-5], that is the higher the Mn content is, the higher the T_t is, the better the damping capacity of the material is. The alloy which contains more than 80 at. % manganese was found to have high damping capacity and optimum strength when quenched from the solid-solution temperature[6]. However, the higher Mn concentration in the alloy would directly cause the alloy more brittle, the lower elongation, impact toughness and the worse corrosion resistance, which will not meet the engineering requirements. The previous research findings revealed that aging treatment under suitable conditions is an effective way to reconcile this problem. *E.g.*, Mn-Cu based damping alloys containing 50-80 at. % Mn can also obtain high T_t and favorable damping capacity by aging treatment in the appropriate temperature range[7]. This is due to the decomposition of γ parent phase into nanoscale Mn-rich regions and nanoscale Cu-rich regions while aging in the temperature range of miscibility gap[8–10], which is considerably beneficial to improve T_t of this alloy along with its damping capacity. Clearly, it is an effectual method which can combine high damping capacity with excellent workability.

M2052 alloy used for forging forming, a representative Mn-Cu based high damping alloy with medium Mn content developed by Kawahara et al.[11], has been extensively studied in the last few decades. Researchers found that M2052 alloy has a good sweet spot among damping capacity, yield strength and workability. Compared with forging technique, casting has been widely used so far due to the simple molding process, low production costs and high productivity *etc*. The influential factors (*e.g.* oscillation frequency, strain amplitude, cooling velocity, heat treatment temperature/time, *etc*.) on damping capacity, microstructure and damping mechanism of M2052 alloy have been studied by some researchers[12-18]. Nevertheless, the casting performance of M2052 alloy is inferior, *e.g.,* a wide range of crystallization temperature, the occurrence of casting porosity, concentrated shrinkage *etc.*, eventually resulting in the unsatisfactory mechanical properties of the castings.

The purpose of this paper is to provide the industrial field with a feasible method of obtaining a cast Mn-Cu based alloy with excellent properties which can be used in machinery and precision instruments industry to reduce vibration and ensure the product quality. According to the effect of alloying elements on phase transformation and casting performance, Al element is considered to reduce the γ phase region and the stability of the γ phase, which can make γ phase more easily transform into γ' phase with micro-twins. Moreover, the solution of Al atoms in the γ phase will increase the strength of the alloy, which can improve the mechanical properties. Also, Al element is one of the important elements which can improve the casting properties of Mn-Cu alloy. Zn element is beneficial to improving the casting and damping properties of the alloy. Finally, 2 wt. % Zn and 3 wt. % Al were added in the MnCuNiFe quaternary alloy in this work and a new cast Mn-26Cu-12Ni-2Fe-2Zn-3Al (wt. %) alloy was developed. Furthermore, several different heat treatment methods are used in this work and their distinct effects are discussed as follows. The homogenization treatment was used to reduce dendrite segregation. The solution treatment was used for impurities immobilization. The aging treatment is used for triggering spinodal decomposition, meanwhile the various aging times for seeking out the optimizing parameters for both excellent damping capacity and high service temperature. Ultimately, a preferable heat treatment method was screened for superior damping capacity as well as high service temperature.

It turned out that the maximum internal friction (Q^{-1}) and the highest service temperature can be achieved concurrently by aging at 435 °C for 2 hrs. Because of the simplicity and efficiency of this preparation method, a novel as-cast Mn-Cu based damping alloy with excellent performance can be produced, which is of important practical significance for its engineering application. This method is particularly suitable for the preparation of casting Mn-Cu based high damping alloy which can be used for vibration reduction.

PROTOCOL:

1. Preparation of Raw Materials

1.1 Weigh all the required raw materials with an electronic scale by mass percentage (65% electrolytic Mn, 26% electrolytic Cu, 2% industrial pure Fe, 2% electrolytic Ni, 3% electrolytic Al and 2% electrolytic Zn), as shown in **Error! Reference source not found.**.

Note: All these raw materials were commercially available.

[insert Figure 1 here]

2. Prepare Metal Casting Mold

Note: The detailed steps of sand casting are shown in [Insert **Figure 1**.

[Insert **Figure 1** here]

2.1 To prepare patterns, make patterns according to the product drawing and make sure that the size of the pattern is expanded to a certain extent to be liable for shrinkage and machining allowances.

Note: The pattern material used in this work is wood (**Figure 3**) because wood pattern is light, easy to work, low cost and of short production cycle.

[Insert **Figure 3** here]

2.2 To prepare the molding sand, mix the quartz sand with 4%-8% sodium silicate to form molding sand.

Note: The sand diameter is about 0.4 mm and the particles are uniform.

2.3 Complete the main molding process by hands.

2.3.1 First, put two patterns in the molding flask.

2.3.2 Then roll over the flask after ramming the molding sand around the patterns and withdraw the patterns from sand.

2.3.3 Finally, brush the surface of the sand mold with casting coating for improving casting surface quality and reducing casting defects.

Note: The molded sand mold is shown in Error! Reference source not found..

2.3.4 Then bake the sand mold in an oven at 180 °C for more than 8 hours before casting to enhance its strength and permeability, facilitate the melt filling and ensure the quality of the casting products.

[Insert **Figure 2** here].

3. Induction 0f Melting

Note: Use a medium frequency vacuum induction melting furnace in the work.

3.1 Open the furnace lid, put 20.8kg Mn, 8.32kg Cu, 0.64kg Ni, 0.64kg Fe, 0.64kg Zn and 0.96kg Al materials in the crucible successively, and cover the materials with cryolite at last.

3.2 Take out the casting mold from the oven and put it in the furnace, adjust its position for successful pouring. Close the lid, vacuum the furnace, and then open the heat distribution system to start melting alloy.

3.3 When the metals start to melt, fill the furnace with argon to 93 KPa negative pressure to inhibit the splashing of the molten metal.

3.4 After the alloy melted, refine it for several minutes to reduce the harmful impurities and gas content.

Note: The melting procedure mainly includes smelting and refining.

4. Casting alloy

4.1 Pour the molten metal smoothly into the casting mold after refining process.

4.2 After the molten metal completely solidified, break the vacuum and take out the casting mold.

4.3 Knock out the castings from casting mold when the temperature of the mold drops to a low level.

5. Pretreatment of castings

The macrophotograph of the molded part is shown in **Error! Reference source not found.**.

5.1 Cut specimens from the casting by using a linear cutting machine.

Note: The specimens for X-Ray diffractometer (XRD) and metallographic observation are in sizes of 10 × 10 × 1 mm^3. The specimens for dynamic thermomechanical analysis (DMA) possess a dimension of 0.8 × 10 × 35 mm^3.

[Insert Figure 3 here]

6. Heat treatment

6.1 Divide the polished specimens into seven groups, among which specimens 1# were free of treatment, maintaining as-cast state for comparison and put others in a box-type resistance oven for different heat treatments.

6.2 Homogenize the specimens 2# and 5# at 850 °C for 24 hrs and quench them in cool water subsequently before aging at 435 °C for 4 hrs and 2 hrs respectively.

6.3 Solution treat the specimens 3# and 6# at 900 °C for 1 hr and subsequently quench them in cool water before aging at 435 °C for 4 hrs and 2 hrs respectively.

6.4 Age the specimens 4# and 7# at 435 °C for 4 hrs and 2 hrs respectively.

7. Damping capacity test

7.1 Use Dynamic Mechanical Analysis (DMA) to measure the damping capacity of the specimens[17].

Note: The test mode is strain sweep at room temperature. During the test, the phase angle δ between the stress and the strain is detected (as shown in **Figure 6**).

7.2 Characterize the damping capacity by Q^{-1}, which can be determined by formula $Q^{-1} = \tan \delta$.

[Insert **Figure 4** here]

8. Sample characterization

8.1 Electrolytic polishing and metallographic observation

8.1.1 For dendrite microstructure observation, etch all specimens for about 1 minute in a mixed solution of perchloric acid and absolute alcohol at 1:27.

8.1.2 Then clean the specimens with acetone, dry the sample with a blower, and observe the dendritic structure with a metallographic microscope.

8.2 Phase structure characterization

Characterize the phase structure and lattice parameters of specimens by X-ray diffraction (XRD) with Cu Kα radiation.

Note: Use a scan speed at 2 °/min. Before XRD measurement prepare the specimens carefully by removing surface stress.

REPRESENTATIVE RESULTS:

[Insert Figure 5 shows the dependency of damping capacity on strain-amplitude for the as-cast MnCuNiFeZnAl alloy specimens 1#-7# and as-cast M2052. The results show that the damping capacity of specimen 1# is higher than that of cast M2052 alloy (as shown in **[Insert Figure 5** (a)) and the traditional forged M2052 high damping alloy mentioned in literatures[20-21]. Moreover, the damping capacity of original as-cast MnCuNiFeZnAl alloy can be further improved by subsequently homogenization-aging, solution-aging and aging treatments (as shown in **[Insert Figure 5**(b), (c)), among which aging treatment for 2 hrs can lead to the highest damping capacity. When the strain amplitude ε is 2×10^{-4}, the Q^{-1} of specimens 1#-7# are listed in [Insert **Table 1**. In addition, it was found that the Q^{-1} can be significantly improved by a shorter aging time compared specimen 4# with specimen 7# (as shown in **[Insert Figure 5** (d)). Furthermore, sand casting and aging for 2 hrs are simpler, economical and efficient compared with forging.

[Insert **Figure 5** here]

[Insert **Table 1** here]

[Insert Figure 6 shows metallographic micrographs of as-cast MnCuNiFeZnAl alloy specimens 1#, 5#-7# respectively. There will form a serious dendritic segregation during the process of slow cooling in the casting molding for the slow diffusion rate of Mn atoms between Cu atoms, which eventually leads to the formation of dendritic microstructure. Since Mn is more susceptible to corrosion than Cu, the dark regions in the observed dendritic structure are Mn-rich dendrites, with a few millimeters long and several micrometers wide, while the light regions are Cu-rich regions. When the temperature decreases, the Mn-rich dendrites mainly precipitate from the liquid phase of the Mn-rich regions and then Cu-rich intervals form between them. By comparison, the dimensions of dark Mn-rich dendrites of specimen 5# are significantly smaller than that of specimen 1#, which indicate that the dendrite segregation of specimen 5# was weakened to some extent. Similarly, the dendrite segregation of specimen 6# was weakened to some extent too, but slightly better than that of specimen 5# due to the shorter holding time during the solution-aging treatment. However, there is no distinctive difference in dendritical microstructure between specimens 7# and specimens 1#. These results represent that the homogenization-aging and solution-aging treatments can weaken the macroscopic Mn segregation, but the direct aging treatment has no obvious effect on it. These conclusions also can be drawn from the compositional EDS analysis. Before spinodal decomposition the Mn content in the Mn-rich dendrites of as-cast MnCuNiFeZnAl alloy was 79.23 at. % on average, and the Mn content was significantly reduced to 68.20 at. % after homogenizing at 850 °C for 24 hrs and 73.42 at. % after solution treatment at 900 °C for 1 hr.

[Insert Figure 6 here]

According to the temperature-dependent damping capacity curve, the damping capacity decreases rapidly as the temperature rises. The temperature at which damping capacity is drastically decreased is usually defined as the service temperature, which is one of the most pivotal indicators for damping alloys being used in engineering area. The service temperatures of specimens 1#, 5#-7# are listed in [Insert Table 2. It can be seen clearly that aging at 435 °C for 2 hrs can cause the optimal service temperature.

[Insert Table 2 here]

The high damping capacity of Mn-Cu based alloy is related to the γ' phase produced in f.c.c-f.c.t martensitic transformation. Normally, the amount of γ' phase is related to Mn content. A large number of scholars[7, 22-24] studied the relationship between lattice parameters, lattice distortion and Mn content in Mn-Cu based alloys. According to the c/a values of specimens 1#, 5#-7#, the Mn content in nanoscale Mn-rich regions of each specimen after spinodal decomposition can be estimated by using the formula mentioned in Ref. 17. The C_{Mn} of specimens 1#, 5#-7# are 84.18 at. %, 84.75 at. %, 85.08 at. % and 85.35 at. % respectively in nanoscale Mn-rich regions after spinodal decomposition. Obviously, the specimen 7# has the highest C_{Mn} which means that the as-cast MnCuNiFeZnAl alloy has the superior damping capacity and higher service temperature simultaneously by aging at 435 °C for 2 hrs.

The relationship among lattice distortion (a/c-1), Q^{-1} (at a strain amplitude of $\varepsilon = 2 \times 10^{-4}$) and service temperature of as-cast MnCuNiFeZnAl alloys subjected to different heat treatments, corresponding to specimens 1#, 5#-7# respectively, is plotted in **[Insert Figure 7**. Evidently, the lattice distortion is directly proportional to the Q^{-1} and service temperature, namely the greater the lattice distortion, the better the damping capacity and the higher service temperature.

[Insert **Figure 7** here]

FIGURE AND TABLE LEGENDS:

Error! Reference source not found.. **Presentation of raw materials** (including 65 wt. %

electrolytic Mn, 26 wt. %electrolytic Cu, 2 wt. % industrial pure Fe, 2 wt. % electrolytic Ni, 2 wt. % electrolytic Zn and 3 wt. % electrolytic Al).

[Insert Figure 1. Steps of sand casting for molding products. The main process includes pattern-making, mold-making and casting operation.

Error! Reference source not found.**. Patterns used in casting mold**. These wood patterns used to obtain the shape of castings.

Error! Reference source not found.**. The molded sand molds**. It has two cavities and its surface has been covered with coating.

Error! Reference source not found.**. The molded parts in the sand mold and the removed parts**. Two castings were molded at one time.

[Insert Figure 4 The fixture construction and testing principle of DMA. (a) Double cantilever fixture of DMA; (b) The relationship of the applied sinusoidal stress to strain and the resultant phase lag. The values of the lag between the stress and strain as well as the modulus can be calculated by formulae.

[Insert Figure 5. The dependency of Q^{-1} on strain-amplitude for the as-cast MnCuNiFeZnAl alloy specimens 1#-7# and as-cast M2052. The test parameters are as follows, For the measurements of strain-amplitude dependence of Q^{-1}, the testing frequency and temperature were 1 Hz and 25 °C, respectively.

[Insert Figure 6**. Metallographic micrographs of as-cast MnCuNiFeZnAl alloys subjected to different heat treatments**. The different dendritic structure of different specimens can be seen from the figure.

[Insert Figure 7 The relationship between lattice distortion (a/c-1), Q^{-1} (ε =2 × 10^{-4}) and service temperature of as-cast MnCuNiFeZnAl alloys subjected to different heat treatments.

[Insert Table 1. The Q^{-1} values of specimens 1#-7# when the strain amplitude ε is 2 × 10^{-4}.

[Insert Table 2**. The service temperatures of specimens 1#, 5#-7#.**

DISCUSSION:

To ensure that this kind of as-cast Mn-Cu based alloy possess both excellent damping capacity and mechanical properties, it is necessary to ensure that the castings have stable chemical composition, high purity and excellent crystal structure. Therefore, strict quality control is necessary in smelting, pouring and heat treatment processes.

Firstly, it is necessary to choose the proper ingredients for the alloy. It would be considered that the added alloy elements can promote the decomposition of γ parent phase, which will help to produce more martensite micro-twins[25]. In addition, certain alloy elements also need to be considered to improve the mechanical and casting properties. The final alloy would combine superior damping capacity and excellent mechanical properties.

Secondly, a reasonable melting process is necessary, which is in connection with the casting characteristics of the alloy. The following key points should be considered in the melting process of cast Mn-Cu based alloys: (1) Feed the metallic raw materials in the crucible in sequence, by adding high-melting-point alloy first, then adding low-melting-point alloy to prevent serious burning loss. (2) Adopt vacuum melting method to ensure that the gas and impurity contents in the alloy are low. At the same time, the inert gas is injected into the furnace to control the pressure and reduce the volatilization of metal liquid during the vacuum melting. (3) When there are no bubbles escaping from the surface of the molten metal, it enters the refining period. The purpose of refining period is to remove gas and volatile inclusions.

The more important step is the choice of heat treatment process. After obtaining the as-cast MnCuNiFeZnAl alloy with excellent performance, a heat treatment process suitable for this alloy is also selected to further improve its damping capacity. Through analyzing the experimental results, it is found that the damping capacity can achieve the extreme value by a short time aging treatment. The final heat treatment process for as-cast MnCuNiFeZnAl alloy is very simple and effective.

Finally, an optimization solution can be achieved for a new cast Mn-22.68Cu-1.89Ni-1.99Fe-1.70Zn-6.16Al (at. %) alloy through investigating the effect of heat treatments on damping capacity and service temperature. That is, the greatest degree of nano-Mn segregation can be achieved by aging at 435 °C for 2 hrs, which result in T_t increasing, eventually significantly improve damping capacity ($Q^{-1} = 5.0 \times 10^{-2}$) and service temperature (70 °C) compared with the original as-cast alloy.

Although this method is just used for casting molding Mn-Cu based high damping alloy, it has the following advantages, such as cheaper modeling materials, simpler mold manufacturing process, higher damping capacity and mechanical property of products, etc. Besides, this method is suitable for different batch of production, both small batch production and mass production. Accordingly, this method is of great significance to improve the effect of vibration reduction and it help to widen the scope of its industry application. It is because of these advantages of this method, it can replace forging technology to produce high-damping products in some areas.

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DISCLOSURES:
The authors have nothing to disclose.

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Figure 1

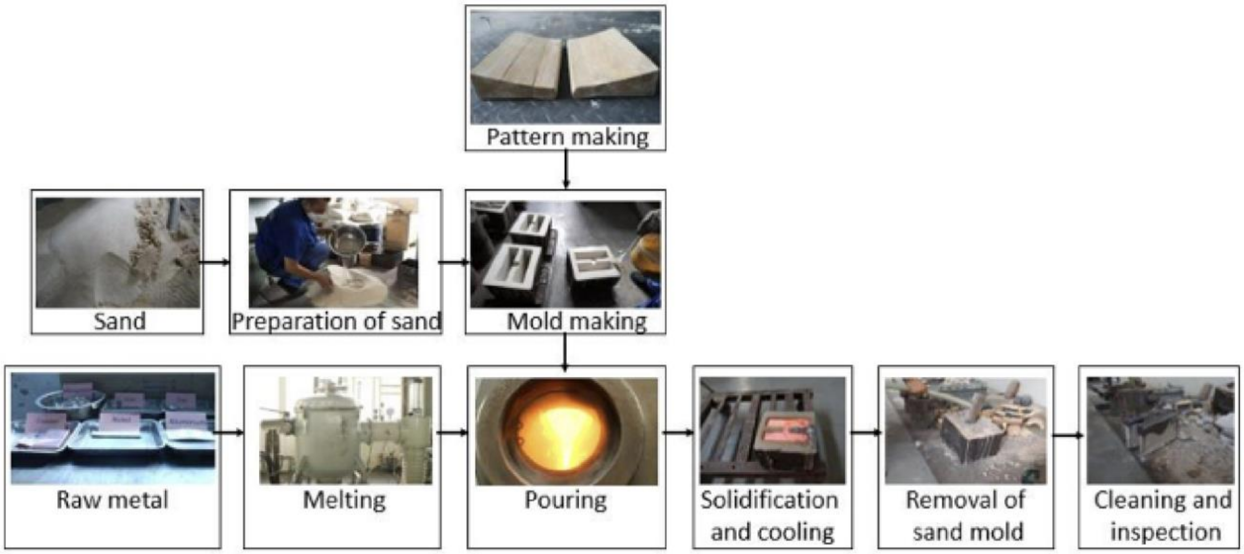
Pattern making
Sand
Preparation of sand
Mold making
Raw metal
Melting
Pouring
Solidification and cooling
Removal of sand mold
Cleaning and inspection

Figure 2

Figure 3

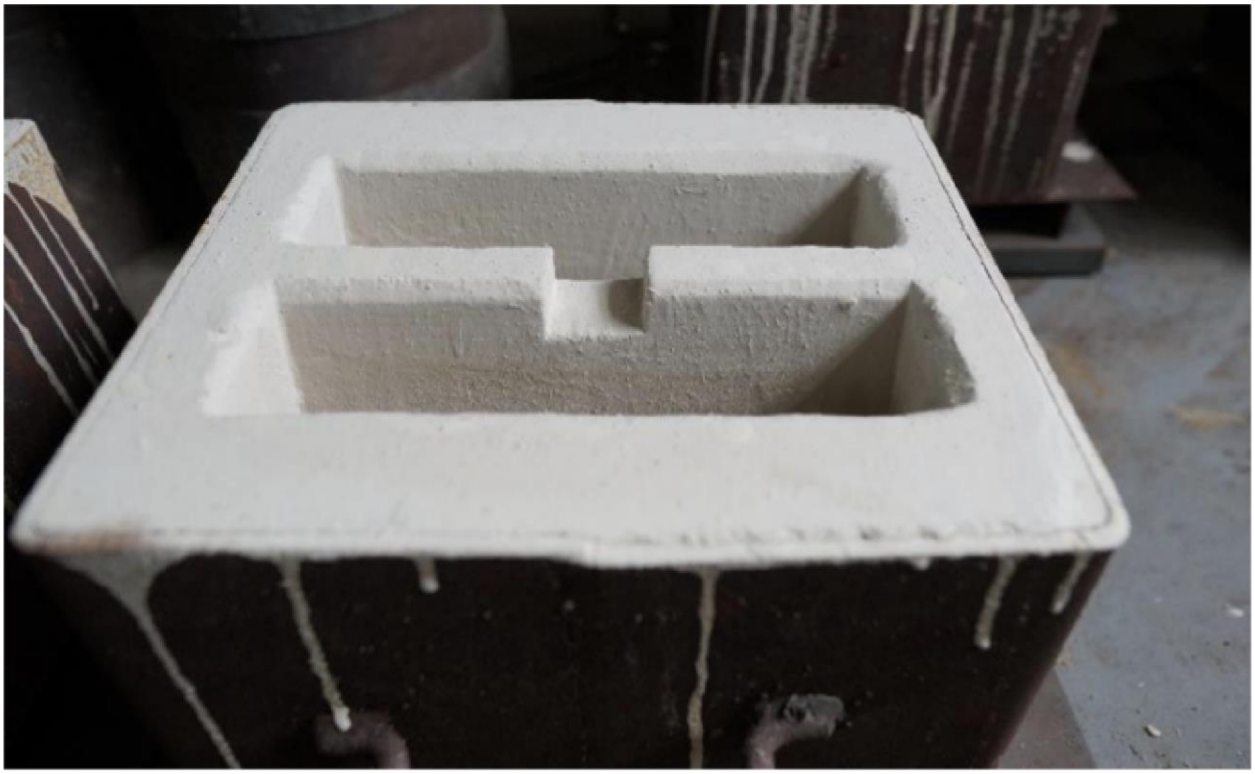

Figure 4

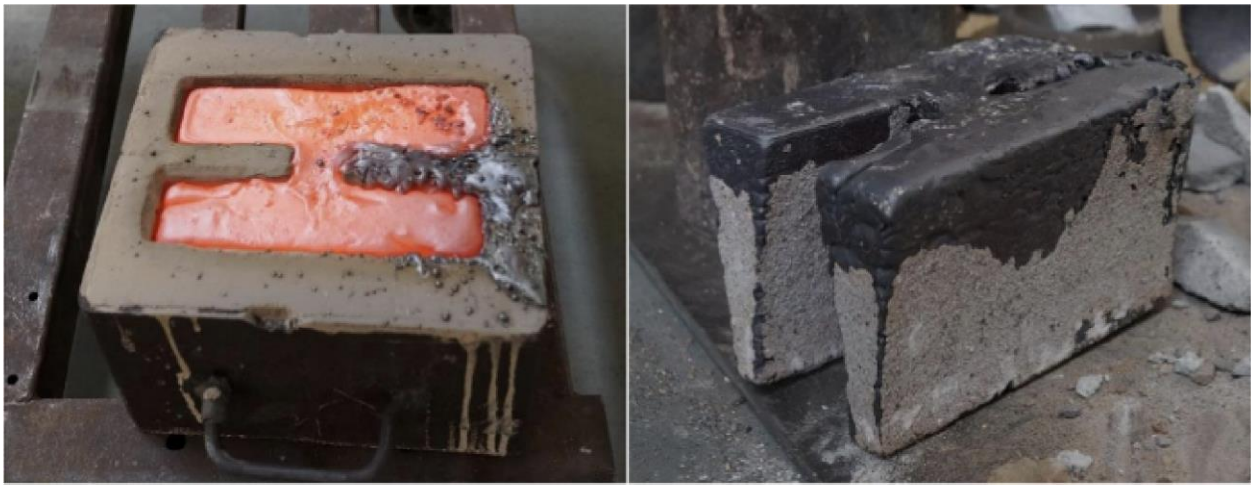

Figure 5

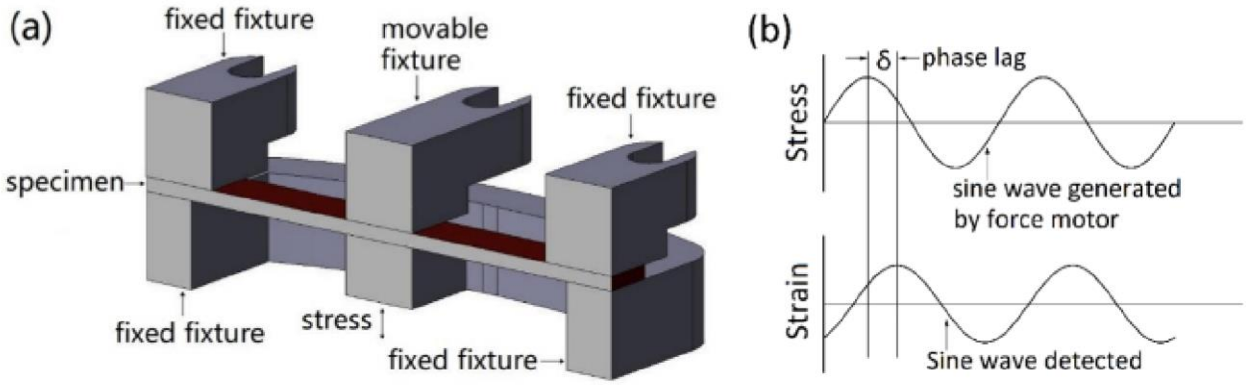
(a)
fixed fixture
movable fixture
fixed fixture
specimen
fixed fixture
stress
fixed fixture
(b)
δ
phase lag
Stress
sine wave generated by force motor
Strain
Sine wave detected

Figure 6

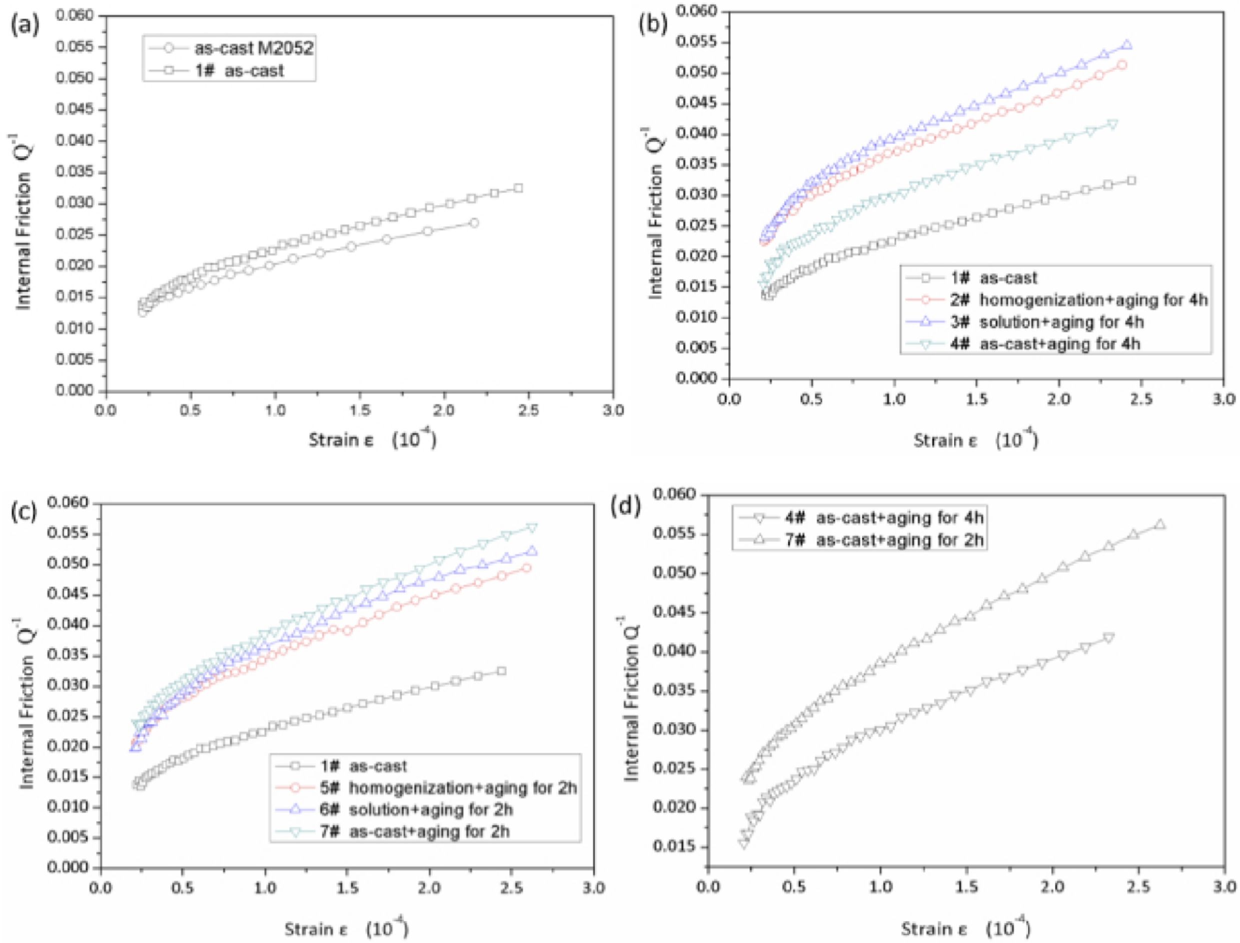
(a)
as-cast M2052
1# as-cast
Internal Friction Q-1
Strain ε (10-4)
(b)
1# as-cast
2# homogenization+aging for 4h
3# solution+aging for 4h
4# as-cast+aging for 4h
Internal Friction Q-1
Strain ε (10-4)
(c)
1# as-cast
5# homogenization+aging for 2h
6# solution+aging for 2h
7# as-cast+aging for 2h
Internal Friction Q-1
Strain ε (10-4)
(d)
4# as-cast+aging for 4h
7# as-cast+aging for 2h
Internal Friction Q-1
Strain ε (10-4)

Figure 7

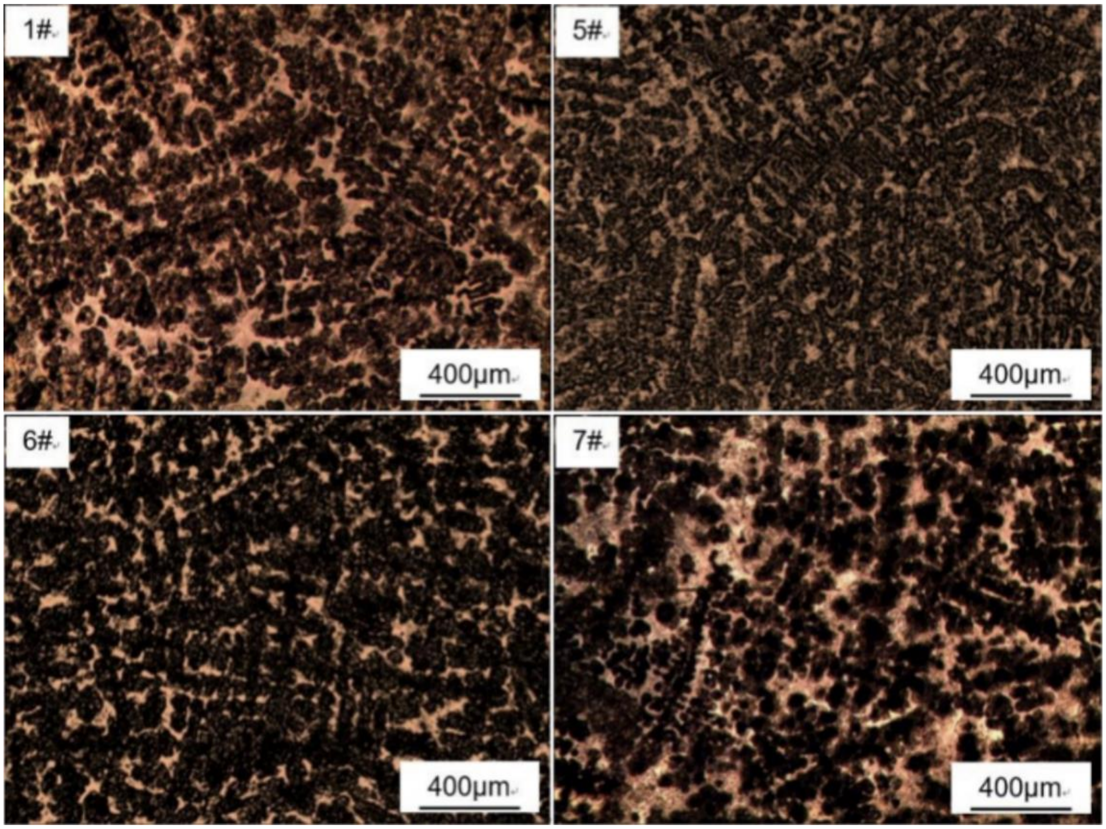

Figure 8

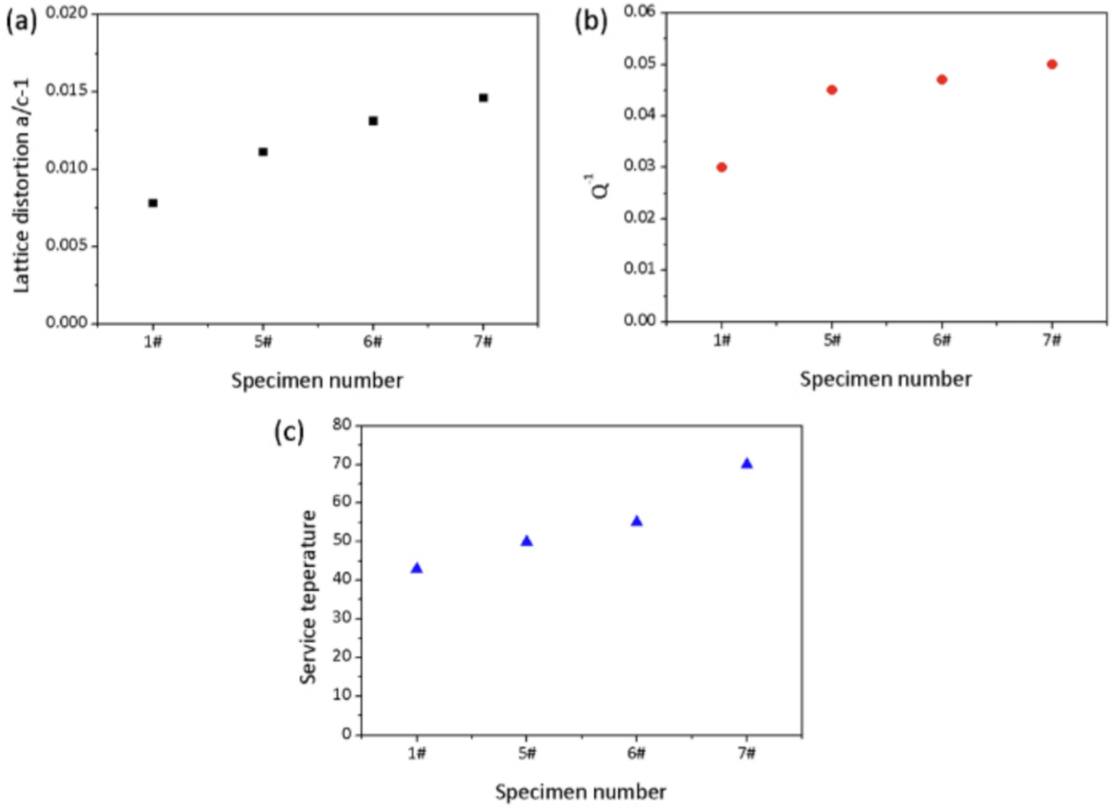
(a)
Lattice distortion a/c-1
0.020
0.015
0.010
0.005
0.000
1#
5#
6#
7#
Specimen number
(b)
Q-1
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1#
5#
6#
7#
Specimen number
(c)
Service teperature
80
70
60
50
40
30
20
10
0
1#
5#
6#
7#
Specimen number

Figure 9

Table 1

Specimens	1#	2#	3#	4#	5#	6#	7#
Q^{-1}	3.0×10^{-2}	4.7×10^{-2}	4.9×10^{-2}	3.9×10^{-2}	4.5×10^{-2}	4.7×10^{-2}	5.0×10^{-2}

Table 2

Specimen	1#	5#	6#	7#
Service temperatures (°C)	43	50	55	70