Technological exploration of preparing hard film on 7A04 aluminum alloy by magnetron sputtering with excellent mechanical properties

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Abstract

In order to improve the quality of hard coatings on the surface of aluminum alloy, the single-stage aging and double-stage aging processes of 7A04 aluminum alloy after solid solution treatment were compared, and the heat treatment process of preparing hard films by magnetron sputtering was explored under the condition of ensuring the mechanical properties of the matrix. After the coating time is determined, the sputtering power is changed, and the best coating method is explored. The experimental results show that, on the premise of the same solution time method, the magnetron sputtering method which limits the coating time and temperature is more suitable than any single-stage aging method. Finally, we have prepared a hard coating with good mechanical properties. The process is: Heat for 60 minutes at 470 ℃, then immediately cool. After three hours at 120 degrees, the film will start to be coated with a power of 210W and sputtering for five hours.

Keywords

7A04 aluminium alloy,aging treatment,microstructure, process exploration.

1. Introduction

7A04 aluminium alloy is one of the most widely used super hard aluminum alloy.But in the practical application, because of the large wear and tear of aluminum matrix, it is very easy to produce wear failure in relative sliding, rotating or vibrating friction system without surface treatment and lubrication, and it is difficult to meet the high requirements of application.[1]Improving the wear resistance of aluminum matrix composites is of great significance for improving their service life and performance. Hard coating on aluminum alloy surface can give full play to the excellent properties of hard coating and aluminum alloy structural parts. Magnetron sputtering has the characteristics of high deposition rate and low substrate temperature rise. It has been widely used in vacuum coating field. In this paper, the preparation of hard film by magnetron sputtering titanium nitride powder on aluminum alloy surface was studied.Deposition of a hard film on the substrate can greatly improve the heat resistance, corrosion resistance and oxidation resistance without changing the composition of the substrate and the original good mechanical properties.However, the hardness of the substrate decreases gradually with the increase of coating time.In order to compensate for this loss, the heat treatment experiments were carried out on the substrate before coating. On the premise of guaranteeing the mechanical properties of the substrate, the optimum technological parameters of preparing hard film on the surface of 7A04 aluminum alloy by magnetron sputtering were explored.

2. Contents and Methods of Experiment

2.1 Experimental Materials

The matrix material is 7A04 aluminum alloy bar, diameter 45mm, long 2mm. The chemical composition of aluminum alloy (wt.%) is shown in Table 1. The heat treatment process is 470 degree solid solution treatment plus artificial aging.

Table 1 Chemical composition of 7A04 aluminum alloy (wt.%)

Material	Zn	Mg	Cu	Cr	Fe	Si	Mn	Al
Content (wt%)	6.24	2.29	1.41	0.11	0.10	0.10	0.20	Bal.

2.2 Experimental Process.

2.2.1 Heat Treatment Experiment.

The sputtering power affects the heating temperature of the substrate during the sputtering process. The output power of 300W is used to sputter the original sample and the solid solution sample for 2, 4, and 6 hours respectively. The measurement results show that the hardness of the substrate decreases.

Therefore, we need to heat the substrate to explore the best heat treatment process before coating.

The specimens were divided into two groups. The first group conducts conventional heat treatment: single stage aging process and two-stage aging process respectively. The single stage aging heating temperature is 120℃ and 140℃ , respectively, and then 2 ~ 20 hours air cooling. In the two-stage aging process, the heating temperature of the first stage aging is 120 ℃, and then air-cooled for 3 hours; the second stage aging process is reheated to 160℃, and then air-cooled for 2 to 8 hours. The aging temperatures of the second group of samples simulate the heating temperatures of the matrix in the vacuum chamber, and the aging temperatures are 180℃, 200℃, 220℃, 250℃, 300℃, and the aging time is determined to be 2 to 10 hours respectively by the limitation of magnetron sputtering time.

2.2.2 Coating Experiment.

Based on the conclusions of the first part of the experiment, the optimum coating temperature and time for obtaining the best hardness are determined.Then we need to change the power of magnetron sputtering to find the best magnetron sputtering process.

2.3 Experimental Method.

2.3.1 High Temperature Solid Solution Treatment.

Firstly, the 7A04 aluminum alloy sample was cut into a cuboid of 10mm×10mm×15mm.After grinding the aged samples one by one, the hardness was measured by Vickers hardness tester. The load was 2.94N, the loading time was 10s. Each sample was measured five times, and the average value was obtained. Next, we will corrode the sample after solution treatment and observe its microstructure by optical microscope. When the corrosion is insufficient, it should be lightly thrown and then etched. When the corrosion is excessive, it should be re-refined and polished. Finally, optical microscopy was used to observe the microstructure.

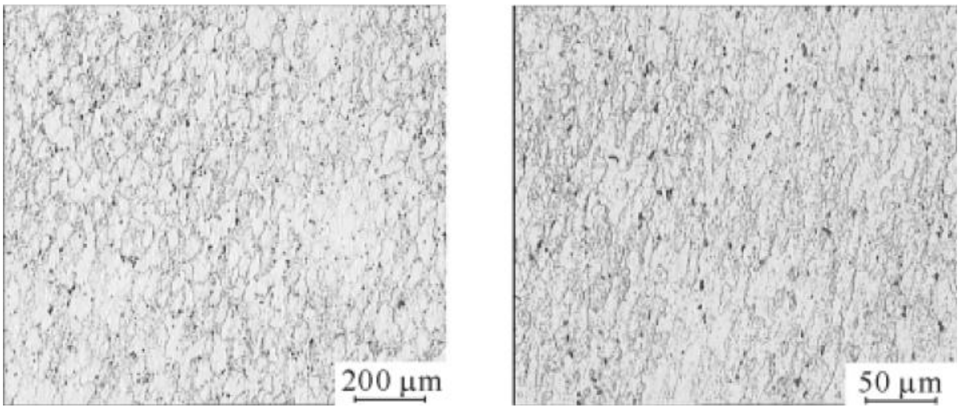
2.3.2 Exploration of Coating Process .

Based on the above experiments, an optimal heat treatment process is obtained. After that, we change the sputtering power and measure the hardness after coating on the premise of fixed sputtering time.

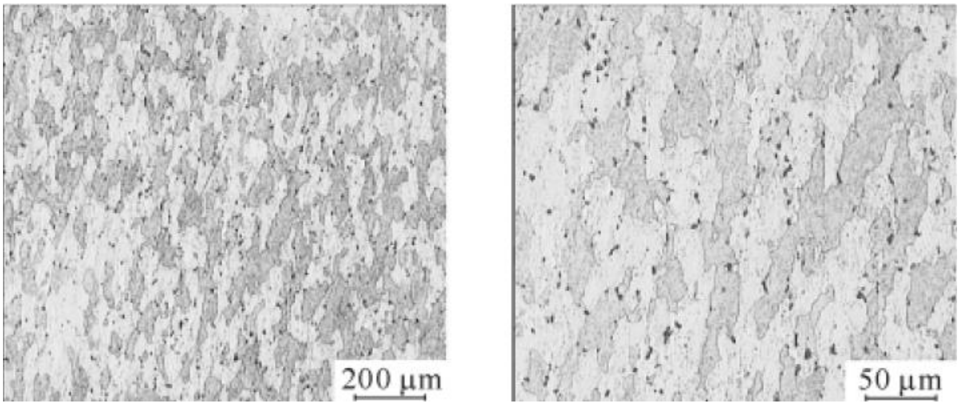
3. Experimental Results and Analysis.

3.1 Microstructure Observation of Original Sample and its Solid Solution .

After grinding, polishing and corrosion, the microstructure of the original sample and the solid solution treated sample was observed by optical microscope. The metallographic structure of the samples before and after solution treatment is shown in Figure 1.



(a)untreated specimens



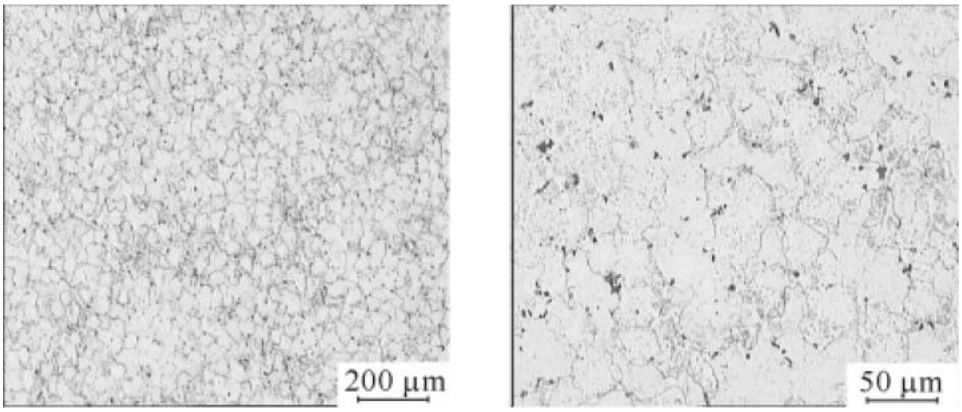
(b) solid solution sample

Fig. 1 Optical micrographs of sample before and after solution treatment

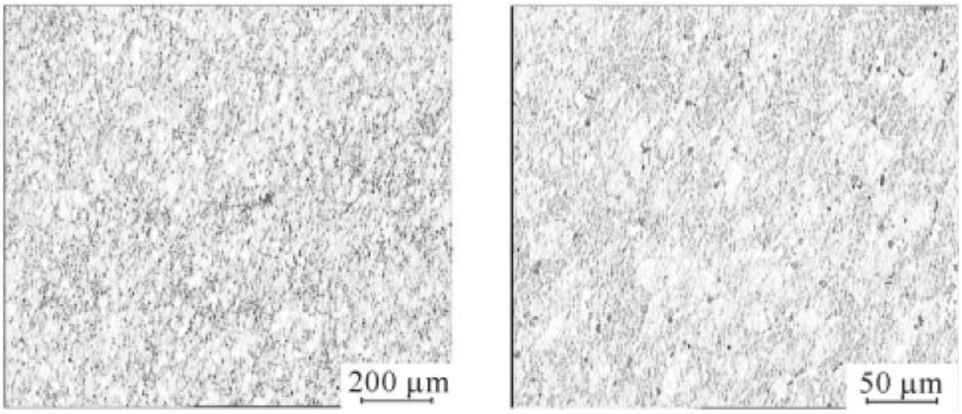
It can be seen from Fig. 1(b) that the solute atoms of the sample heated at 470℃ for one hour gradually dissolve and the supersaturated solid solution with uniform grains can be obtained, which provides a good matrix structure for aging treatment. Thermodynamic analysis shows that the higher the content of solute atoms in the matrix, the greater the driving force of precipitation of strengthening phase during aging treatment;the more fully the phases are dissolved in the aluminum alloy at the appropriate solution temperature and time, the more appropriate the supersaturation of the matrix after solution, and the larger the volume fraction of precipitation of the second phase during aging treatment,Which will increase the hardness and strength of the alloy.

3.2 Microstructure Observation after Aging.

Fig. 2(a) is a metallographic picture of the sample aged at 140℃ for 16 hours. Fig. 2(b) is a metallographic picture of the sample aged by stages.



(a) Single Stage Aging



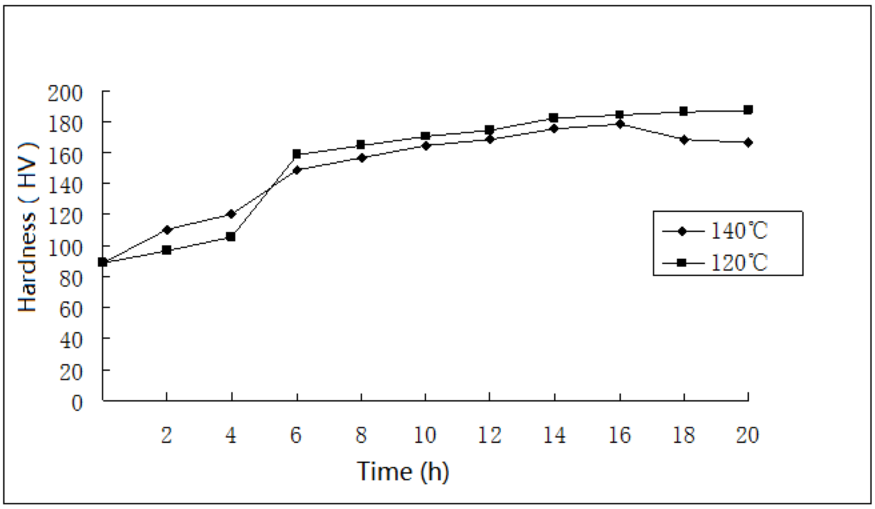
(b) Graded Aging

Fig.2 Optical micrographs of sample at different aging process

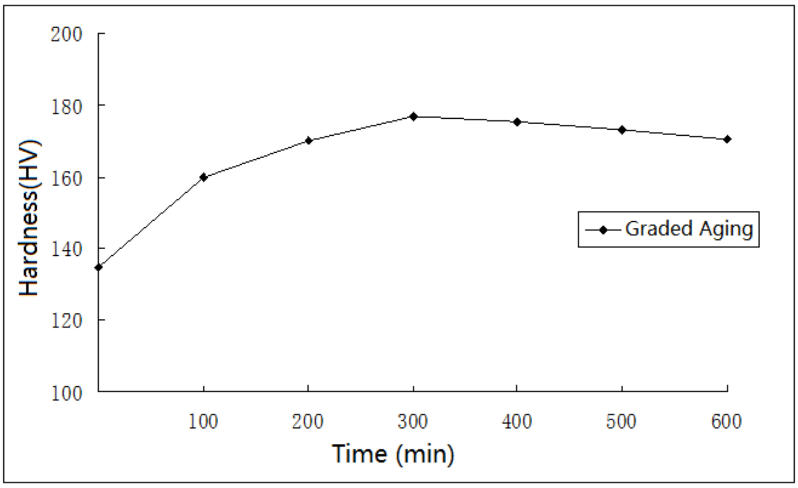
As can be seen from Fig. 2(a), the precipitation sequence of 7A04 aluminum alloy after single-stage aging treatment is: α→GP→η' (Mg-Zn_2)→η($MgZn_2$)→ T ($Al_2Zn_3Mg_3$) [3].According to the aging kinetics theory, the strengthening phases of 7A04 alloy after aging treatment are mainly solute atom segregation (GP) zone and dispersed transition phase η', and the number of stable phaseηis small and concentrated at grain boundaries. The mechanical properties of the composites are mainly determined by the precipitated phase of the matrix. The better mechanical properties can be obtained by adding uniformly dispersed phase and uniformly distributed GP region[4]. The precipitation process is closely related to the morphology, quantity and size of precipitates. The final mechanical properties may be the result of several strengthening mechanisms. It can be seen from Fig.2(b) that light gray Al_6(MnFe) solid solution and dark brown residual S phase, T phase and insoluble impurity phase are distributed on the matrix of α solution after mixed acid etching. Trace elements such as Mn and Cr can form intermetallic compounds and exist as dispersed particles, which can refine grains and effectively prevent grain growth.

3.3 Hardness Testing after Aging.

The hardness changes of 7A04 aluminum alloy after artificial aging at different temperatures and time are shown in Figure 3.



(a) Single Stage Aging



(b) Graded Aging

Fig.3 The hardness contrast between different aging process

As can be seen from Fig. 3(a), the hardness increase is the biggest when the single-stage aging treatment, 120 ℃aging temperature for 6 hours. From the 6th hour, the hardness of the samples treated at 120 ℃ and 140 ℃ increased evenly, indicating that the precipitation rate of the strengthened phase was uniform. When the aging time is prolonged to 20 hours, the aging temperature of 120 C is still underaged, and the hardness will be further increased with time. The hardness of the alloy reaches the highest at sixteenth hours at 140 aging temperature. The hardness decreases with the increase of time, which is the overaging time.

In the stepwise aging experiment, the samples after solid solution treatment were kept for 3 hours at 120 ℃ for air cooling, and then reheated to 160 ℃ for 2 to 8 hours of secondary aging. It can be seen from the hardness curve of step aging in Figure 3 (b) that the hardness of the sample reaches its peak value at the fifth hour, and the hardness decreases slightly compared with that of single-stage aging but remains basically flat, and then decreases with time. Therefore, the maximum hardness of the alloy is fifth hours for peak aging time.

The second group of samples were tested in a simulated vacuum chamber. Because the sputtering power will affect the substrate heating temperature, too small power will lead to poor adhesion between the film and the basic factors, so the set power should not be too small. Considering the possible heating temperature of the substrate during sputtering, the heat treatment experiment of single stage aging temperature of 180℃, 200℃, 220℃, 250℃ and 300 ℃ was carried out, and the coating time was limited to 2 to 8 hours. The hardness curve is shown in Figure 4.

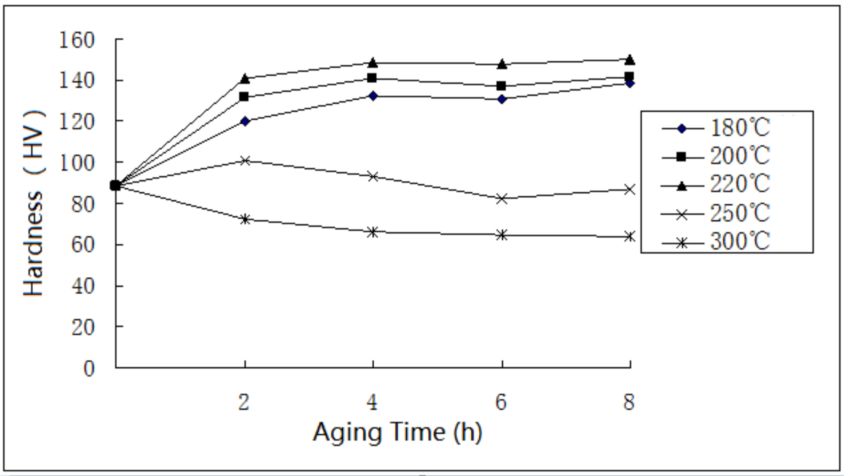


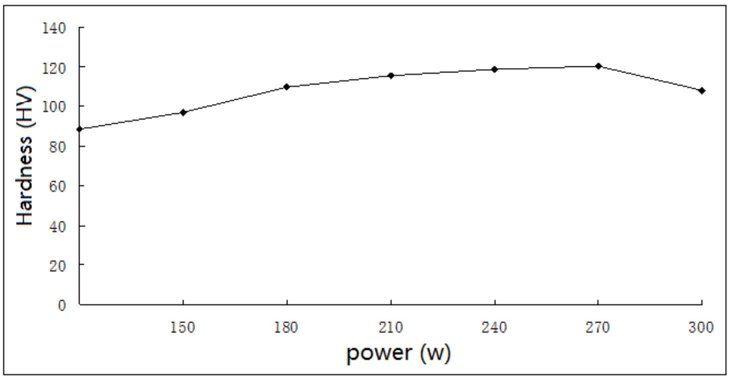
Fig. 4 Hardness curve of 7A04 at different aging temperature

It can be seen from Fig. 4 that the hardness decreases with aging time of 2 to 8 hours at 300 ℃and 250 ℃, and increases slightly at 180℃, 200℃ and 220 ℃. According to the aging kinetics, the diffusion of solute atoms controls the aging process, and the most important factor affecting the atomic diffusion coefficient is the aging temperature. According to the Arrhenius formula: D=D0exp(-Q/RT), the higher the aging temperature T, the larger the diffusion coefficient of solute atoms, the faster the decomposition rate of supersaturated solid solution, the shorter the time to reach the aging peak, and the faster the over-aging rate. The precipitation rate of strengthening phase at ageing temperature of 220℃ is faster than that at 180℃, and the peak ageing time is shorter. When the aging temperature is 300℃, the solute atoms will not form segregation phase, GP region will not form, but directly precipitate transition phase.

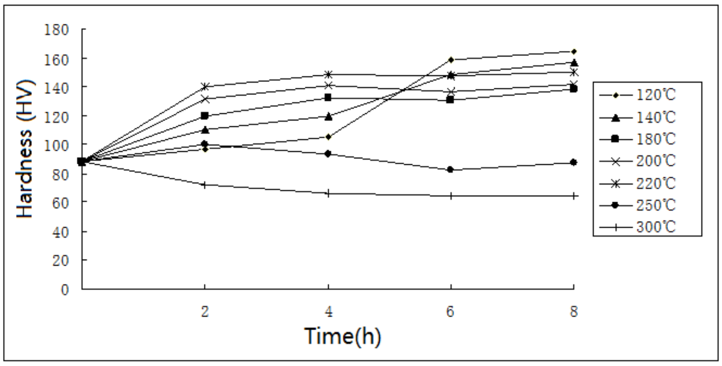
The strength and hardness of the alloy decrease obviously because the transition phase changes rapidly into the coarse equilibrium phase and completely breaks away from the coherent relationship with the matrix. In conclusion, the hardness of the sample does not rise back to the ideal state when the simulated single-stage aging temperature is in the range from 180 ℃to 300 ℃ within the time limit of 2 to 8 hours

3.4 Coating Process

The results show that the highest hardness of 7A04 aluminum alloy treated by single-stage aging is also lower than that of the original sample within the limited time of 2 to 8 hours. In order to ensure the mechanical properties of the substrate, a two-stage aging process was used to coat the substrate. Before the coating, the substrate was treated by the first-stage aging treatment at 120 C for 3 hours, and the second-stage aging treatment time was the coating time at 160 C for 5 hours. In order to explore the suitable power at 160 C, the sputtering time is fixed for 2 hours, and the output power is changed, 150, 180, 240, 270, 300W. The hardness measurement results are shown in Figure 5.



(a) The hardness contrast curve under the same time with different power



(b) The hardness contrast curve under the different time and aging temperature

Fig. 5 Hardness comparison curve

It can be seen from Fig. 5(a)that the effective aging time is 150-270W and the hardness decreases when the power is 300 W, which indicates that the aging temperature is too high. Comparing with the hardness curves obtained before aging at different temperatures as showen in Fig. 5(b), it can be judged that the hardness value of 2 hours output 210W power is between 140 ℃ and 180 ℃ aging temperature, about 160 ℃. It can be determined that the optimum sputtering power is 210W and the time is 5 hours.

4. Conclusion

(1)The results show that the graded aging treatment is more suitable for the preparation of hard film by magnetron sputtering than single aging treatment.

(2) The optimum coating process is: holding at 470 ℃ for 60 minutes, then water cooling, then holding at 120 ℃ for 3 hours. The best coating process is 210 W output power for 5 hours sputtering time. The adhesion between the film and the substrate is good and the hardness of the substrate is almost no loss.

Acknowledgements

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