Effect of two-stage aging on hardness and electrical conductivity of as-extruded 7075 aAluminum Alloy

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Abstract. The effect of two-stage aging on the microstructure and properties of as-extruded 7075 aluminum alloy was studied by means of hardness, conductivity and microscope. The results show that: in the process of solution and primary aging, with the increase of heat treatment time, the hardness and electrical conductivity of the alloy have little improvement, but there is a strong linear correlation between hardness and electrical conductivity, that is, the change of hardness and electrical conductivity presents the same trend; after two-stage aging treatment, with the extension of aging time, the hardness of the alloy first increases, reaches the peak value, and then decreases gradually, the hardness and conductivity have a strong negative linear correlation; and with the increase of aging temperature and aging time, the hardness of the alloy decreases by 9.6%, while the conductivity increases by 18.6%.

1. Introduction
The ultra-high strength aluminum alloy used in aerospace industry has the characteristics of lightweight, precision, large-scale, integrated, uniform microstructure and properties[1]. According to the performance characteristics and performance requirements of aluminum alloy used in various structural parts of aerospace and aircraft, the commonly used high-performance aluminum alloys are: high-strength aluminum alloy, heat-resistant aluminum alloy, corrosion-resistant aluminum alloy. Among them, high strength aluminum alloy is mainly used in the structure of aerospace, aircraft structure, engine room, seat structure, carrier of control transmission system, etc. With the rapid development of aerospace industry, the requirements for comprehensive properties of aluminum alloys, such as strength, hardness, conductivity, etc., are becoming higher and higher. As an important means of material nondestructive testing, the research on conductivity is paid more and more attention, which many researchers pay attention to the fact that its electrical conductivity can not only measure the thermal conductivity level and stress corrosion of alloy, but also the electrical conductivity measurement is relatively simpler than the thermal conductivity and corrosion resistance test. According to the relevant research, when the composition of the alloy is determined, its conductivity depends on the structure of the material itself, and the structure of the material depends not only on the forming method, but also on the heat treatment process[2,3].

7075 aluminum alloy, with the advantages of high strength, low density, good hot workability, good corrosion resistance and toughness, is widely considered as the most important structural material in aerospace and flight, and has become one of the hot spots in the development of structural materials in
the world\cite{4-8}. In the actual industrial production, the heat treatment status of aluminum alloy is generally checked by detecting the conductivity of aluminum alloy. That is to say, the formation and growth of precipitates during the strengthening treatment of aluminum alloy will improve the strength of the material, and these precipitates will also affect the position and movement of electrons and reduce the conductivity of the material, and these precipitated phases will also affect the position and movement of these electrons, which reduce the electrical conductivity of the alloys, and then control the quality of the aluminum alloy the heat treatment process to solve the overheating problem that may be encountered in the manufacturing and maintenance of aircraft skins and structural parts\cite{9}. The strength and hardness of 7075 aluminum alloy have obvious aging bimodal characteristics, but the best aging heat treatment process does not necessarily get qualified conductivity. The annealing temperature and annealing holding time, solution temperature and solution holding time, cooling rate and sampling method have important effects on the conductivity. In the actual production, it is difficult to ensure that the strength, hardness and other mechanical properties and electrical conductivity indicators can meet the standard requirements, so optimizing the heat treatment system is an important measure to improve the comprehensive properties of high strength aluminum alloy in the corresponding fields. In this paper, commercial extruded 7075 aluminum alloy is used as the research carrier, and the optimal heat treatment process of 7075 aluminum alloy is discussed and analyzed through the orthogonal experiment of two-stage solid solution and two-stage aging method, so as to ensure that the mechanical properties and electrical conductivity of 7075 aluminum alloy can meet the standard requirements, and lay a theoretical foundation for the study of corrosion resistance of 7075 aluminum alloy.

2. Experimental materials and methods

2.1 preparation of experimental materials
Commercial as-extruded 7075 aluminum alloy with a diameter of 25mm is selected as the experimental material, and its composition is shown in Tab.1. The alloy was treated by two-stage solid solution and two-stage aging treatment, and the Rockwell hardness of the experimental samples was tested, and the average value of 10 points was taken for each sample; the conductivity of the samples was tested by PZ-60A vortex conductivity meter, and the average value was obtained by measuring 5 times at different positions of the samples; after grinding and polishing, the samples were corroded by Keller reagent, and the results were analyzed by optical microscopy. the effect of heat treatment on the relationship between microstructure, hardness and electrical conductivity was analyzed.

| Element | Zn  | Mg  | Cu  | Cr  | Mn | Si | Fe  | Ti  | other |
|---------|-----|-----|-----|-----|----|----|-----|-----|-------|
| national standard | 5.1~6.1 | 2.1~2.9 | 1.2~2.0 | 0.18~0.28 | $\leq$ | $\leq$ | $\leq$ | 0.2 | $\leq$ $\leq$ |
| Actual measurement | 5.63 | 2.56 | 1.54 | 0.23 | 0.08 | 0.17 | 0.28 | 0.08 | 0.05 $\leq$ |

2.2 Experimental Process and Method
In this experiment, the effect of two-stage solution aging and two-stage aging on the hardness and conductivity of extruded 7075 aluminum alloy was analyzed and studied

(1) Determination of solution treatment process scheme: the first solution temperature of 7075 aluminum alloy with diameter of 25 mm is 470 °C, and the solution time is 30 min, 1 h, 2 h and 5 h respectively; the hardness and conductivity of 7075 aluminum alloy are shown in Table 2 with water cooling at 60 °C.
| Tab. 2 Hardness and Conductivity of 7075 aluminum alloy after solution treatment at 470 ℃ |
|-----------------------------------------------|----------------|----------------|
| Holding time(h) | Hardness(HRB) | Conductivity(%IACS) |
| 0               | 61.3          | 39.8           |
| 0.5             | 17.8          | 42             |
| 1               | 20.8          | 43             |
| 2               | 19.1          | 41.6           |
| 5               | 18.4          | 41.5           |

According to the experimental results, the conductivity of 7075 aluminum alloy increased by 8%, but its hardness decreased significantly. The comprehensive indexes of hardness and conductivity were better. The first solution process was 470℃ × 1h, water cooling at 60℃. On this basis, the second solution treatment was carried out on 7075 aluminum alloy bar. The temperature was 480 ℃, and the solution holding time was 0.5h, 1h, 2h and 3h respectively. The hardness and conductivity are shown in Tab.3

| Tab.3 Hardness and conductivity of 7075 aluminum alloy after second solution treatment(480℃) |
|-----------------------------------------------|----------------|----------------|
| Holding time(h) | Hardness(HRB) | Conductivity(%IACS) |
| 0.5             | 18.7          | 41.8           |
| 1               | 18.5          | 44.5           |
| 2               | 17.7          | 43.1           |
| 3               | 17.1          | 42.8           |
| 4               | 17.5          | 41.9           |

According to the experimental measurement of the second solid solution process, the comprehensive index of hardness and conductivity of the alloy after 480℃ × 1h and 45℃ water cooling is the best, so the second solid solution process is 480℃ × 1h and 45℃ water cooling.

(2) Two-stage aging heat treatment process scheme: the main purpose of this experiment is to study the 7075 aluminum alloy to meet the standard of electrical conductivity while meeting the mechanical properties. Therefore, the alloy is subject to two-stage aging process, and the process scheme is shown in Tab. 4

| Tab.4 Hardness and conductivity of 7075 aluminum alloy after primary aging |
|-----------------------------------------------|----------------|----------------|
| Holding time(h) | Hardness(HRB) | Conductivity(%IACS) |
| 1               | 23.3          | 39.9           |
| 2               | 26.7          | 40.1           |
| 4               | 28            | 42             |
| 6               | 35.9          | 44.8           |
| 8               | 33.3          | 42.8           |
| 10              | 31.3          | 41.2           |
The best comprehensive indexes of hardness and conductivity of 7075 aluminum alloy after primary aging are 35.9 HRB and 41.8% IACS, as shown in Table 4. The aging process is 120°C × 6h, water cooling at room temperature. On this basis, the 7075 aluminum alloy is subjected to secondary aging at 160°C, 180°C and 200°C. The process scheme is shown in Tab. 5

| Holding time (h) | 160°C | 180°C | 200°C |
|------------------|-------|-------|-------|
|                  | Hardness (HRB) | Conductivity (%IACS) | Hardness (HRB) | Conductivity (%IACS) | Hardness (HRB) | Conductivity (%IACS) |
| 1                | 48.7 | 42.5 | 45.5 | 44.5 | 36.2 | 45.2 |
| 2                | 46.9 | 43.2 | 55.4 | 41.3 | 37   | 44.9 |
| 4                | 51   | 41.2 | 46   | 44.2 | 39.2 | 44.4 |
| 6                | 39.5 | 45.8 | 43.9 | 44.8 | 37.7 | 44.9 |
| 8                | 46.3 | 43.2 | 40.9 | 45.5 | 37.9 | 44.9 |
| 10               | 47.5 | 42.9 | 43.1 | 44.9 | 37.3 | 45   |
| 12               | 48.2 | 42.5 | 46.9 | 44   | 37.9 | 44.9 |
| 14               | 49.2 | 41.7 | 46.9 | 44.1 | 36.3 | 45.5 |
| 16               | 47   | 43.1 | 39   | 47.2 | 32.9 | 47.1 |
| 18               | 42.2 | 44.3 | 44.1 | 44.3 | 36.7 | 45.4 |
| 20               | 44.2 | 43.6 | 39.3 | 46.2 | 36.2 | 45.6 |
| 22               | 44.9 | 43   | 39.4 | 45.9 | 37.4 | 46   |
| 24               | 41.2 | 44.3 | 40.1 | 45.8 | 31.7 | 47.4 |

3. Experimental results, discussion and analysis

3.1 Effect of solution process on hardness and conductivity of 7075 aluminum alloy bar

The influence of two-stage solution process on the hardness and conductivity of 7075 aluminum alloy is shown in Tab.1~2. It can be seen from Tab.1 and Tab. 2 that with the extension of holding time, the hardness and conductivity of 7075 aluminum alloy bar first increase and then decrease, and the change trend of conductivity and hardness is the same. According to the relevant research\(^9\), the conductivity of the alloy can be used as the evaluation basis for homogenization treatment. With the increase of solution holding time, the second phase precipitates in the alloy structure, and the size of insoluble phase also decreases, resulting in the change of microstructure or diffusion migration of local components. These changes will affect the conductivity of the alloy. Therefore, through the determination of conductivity, the conductivity of the alloy can be improved To estimate the change of internal structure of the alloy and optimize it. The peak values of electrical conductivity and hardness of extruded 7075 aluminum alloy are 20.8 HRB and 43% IACS after primary solution and 18.5 HRB and 44.5% IACS after secondary solution. The change trend of electrical conductivity and hardness of the alloy is the same, that is, when the electrical conductivity reaches the peak, the internal homogenization degree of the alloy is the best. After two-stage solution treatment, the electrical conductivity of the alloy increases by 11.8%, but the hardness decreases greatly. The main reason is that the higher the solution temperature, the larger the grain size of the alloy, the lower the density of the alloy, and the lower the strength and hardness, in addition, the number of solute atoms dissolved in the alloy is reduced, the lattice distortion of the matrix is weakened, and the free path of electrons is increased, which makes the electrical conductivity of the alloy improved obviously. Therefore, the best solution treatment process is 470°C × 1h (60°C water cooling) +480°C × 1h (45°C water cooling).
3.2 Effect of two-stage aging on hardness and conductivity of 7075 aluminum alloy bar

3.2.1 Effect of bipolar aging on the conductivity of 7075 aluminum alloy hardness tester. According to the relevant research\cite{11}, the conductivity and hardness of alloy are random variables, which are affected by many random factors, and the influence of each random factor does not play a decisive role, but these factors can be superimposed. The limit theory in probability theory holds that these random factors are generally considered to obey the normal distribution \( N(a, \sigma^2) \) with continuous density function of

\[
\phi(x) = \frac{1}{\sqrt{2\pi}} e^{-\frac{(x-a)^2}{2\sigma^2}}, \quad (a \text{ and } \sigma^2 \text{ are constants greater than zero}).
\]

The relationship between the related factors can be evaluated according to advantages and disadvantages of point estimation and interval estimation of mathematical statistics and the correlation coefficient:

\[
\rho(x, y) = \frac{\sum_{i=1}^{n} (x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum_{i=1}^{n} (x_i - \bar{x})^2 \cdot \sum_{i=1}^{n} (y_i - \bar{y})^2}} = \frac{n \sum_{i=1}^{n} x_i y_i - \sum_{i=1}^{n} x_i \cdot \sum_{i=1}^{n} y_i}{\sqrt{n \sum_{i=1}^{n} (x_i)^2 - (\sum_{i=1}^{n} x_i)^2} \cdot \sqrt{n \sum_{i=1}^{n} (y_i)^2 - (\sum_{i=1}^{n} y_i)^2}}.
\] (1)

After one-stage aging, the peel correlation coefficient between hardness and conductivity of 7075 aluminum alloy is \( \rho(x, y) = 0.89823 \), which belongs to the positive strong linear correlation, that is, with the increase of hardness, the maximum value of conductivity increases, as shown in Fig. 1. After one-stage solid solution, the conductivity and hardness of the alloy were tested at different aging time. With the increase of holding time, the conductivity and hardness of the alloy first increased and then decreased, as shown in Fig.2. The main reason is that the solute atoms in the supersaturated solid solution gradually precipitate with the prolongation of aging holding time, which reduces the degree of lattice distortion in the alloy, reduces the internal stress, and increases the free path of free electrons in the alloy. Therefore, the conductivity and hardness of the alloy will gradually increase, but the improvement of conductivity is more obvious than that of hardness. The more homogeneous and sufficient the precipitation of the aged strengthening phase is, the higher the electrical conductivity of the alloy will be. When the time effect reaches a certain stage, the hardness and electrical conductivity of the alloy will decrease or change little. The main reason is that the longer the precipitation of the strengthening phase, the larger the grain size and strengthening phase of the alloy, which increases the probability of electron backscattering and makes the hardness and electrical conductivity of the alloy better. The conductivity will decrease.

Fig. 1 Relationship between conductivity and hardness after primary aging
Fig. 2 Change curve of conductivity and hardness under different holding time

After two-stage solid solution, 120°C×6h+(160°C, 180°C, 200°C) was used to select different aging temperature and holding time, and the hardness and conductivity of samples were tested. The curve of conductivity and hardness changing with the increase of holding time was obtained, as shown in Fig. 3, the hardness of the alloy increased with the extension of holding time in wavy manner, and then after reaching the peak, while the conductivity decreased. In the two-stage aging process of 180°C, the hardness of the alloy gradually increases with time before holding time of 2h, while the conductivity gradually decreases. The maximum hardness of the alloy reaches 55.4HRB at 180°C×2h, and the conductivity corresponding to the peak hardness is the lowest among the three processes, which is 41.3% IACS. After holding time of 2h, the hardness decreases with extension holding time, while the conductivity of increases with the extension of holding time. When the holding time is 16h, the peak value of conductivity reaches 47.2% IACS, while the corresponding hardness value is the lowest, which is 39HRB. After aging for 16h, the conductivity of 7075 decreases obviously, but the hardness of 7075 does not changes little, as shown in Fig.3(b). With the increase of aging temperature, the hardness of the alloy increases first and then decreases. In the 200°C aging process, the highest hardness is 39.2HRB, the lowest is 31.7HRB, and the highest conductivity is 47.4%IACS, the lowest is 44.4%IACS. The overall conductivity is improved to some extent, but the hardness of the alloy decreases significantly, as shown in Fig. 3 (c), so it is not valuable as a structural material.

According to the three process schemes shown in Fig.3, the change trend of conductivity and hardness is basically opposite. According to pear correlation coefficient formula (1), the correlation coefficients between conductivity and hardness are -0.96962, -0.94344, -0.86689 in the three secondary aging temperature process schemes, which are all negative values and belonging to the negative strong linear correlation, that is, with the increase of hardness, the conductivity of the alloy decreases, as shown in Fig. 4. Comprehensive analysis shows that after 470°C×1h (60°C water cooling) + 480°C×1h (45°C water cooling) + 120°C×6h (water cooling) + 180°C×2h (water cooling) heat treatment, its conductivity is increased by 18.6% compared with the original state, but its hardness is decreased by 9.6%. Therefore, it will sacrifice the mechanical properties of the alloy at the same time to obtain high conductivity.
The microstructure of 7075 aluminum alloy under different heat treatment process is shown in Fig.5. There are more uniform black round punctate precipitates with a small amount of agglomeration in Fig.5(a), and the gray part is the matrix of 7075 alloy. After two-stage aging treatment, the second phase of Fig.5(b)–(c) and Fig.5(e)–(g) are coarsening and growing up obviously, and they all gather in different degrees. The black or gray spherical or short rod-shaped second phase precipitated in the alloy may be η' and η strengthening phase, and there are more discontinuous spherical precipitates and wider grains boundary non precipitation area. These microstructures seriously reduce the mechanical properties of the alloy, but maintains a high conductivity. the second phase in Fig.5(d) is mainly spherical and evenly distributed, the second phase has less aggregation and growth particles, and the continuous degree of grain boundary precipitation is higher. The main reason is that under the condition of the second aging temperature of 180℃, the aging time is short, which makes the η' phase and η phase have a small tendency to coarsen and grow, so that the precipitation phase spacing does not change much, so its hardness value is relatively high, but lower than the original 7075 alloy, and its electrical conductivity reaches 41.3%. With the prolongation of aging holding time, the size of precipitated phases increased obviously, and there are obvious aggregation, coarsening and other phenomena, so the hardness value of the alloy decreases greatly, but its conductivity increases by 18.6%.
3.2.2 Analysis and discussion. Aging heat treatment is one of the important means to improve the properties of 7075 aluminum alloy. It is easy to get an unstable supersaturated solid solution during solution heat treatment, and the unstable supersaturated solid solution will decompose during aging heat treatment. In the process of aging heat treatment, due to the different aging temperature and aging time, the degree of solid solution decomposition and precipitated phase are also very different. It is generally believed that the aging precipitation sequence of ultra-high strength aluminum alloy is: α supersaturated solid solution → GP area → η′ transition phase (MgZn2) → η equilibrium phase (MgZn2). Since the final stable equilibrium phase η is not coherent with the matrix, its interface energy is high and cannot be directly precipitated from the matrix. Therefore, in the two-stage aging process, the first stage aging is carried out at low temperature. The purpose is to form a certain amount of GP region which is more than the critical size and is consistent with the matrix, and the GP region is most likely to precipitate η′ phase which is semi-coherent with the matrix. In the second aging process, with the prolongation of aging time, the critical size GP region formed in the low temperature aging stage will act as the nucleation core of η′ phase, and then the semi-coherent transition η′ phase will gradually precipitate, and then the transition η′ phase will gradually gather and grow into stable equilibrium η phase. When there are more and smaller precipitation strengthening, the alloy matrix will have obvious lattice distortion, resulting in aging strengthening effect, but it will also increase the free electron scattering, lead to the decrease of conductivity. When the aging time continuous to increase, the precipitated phase in the alloy matrix gradually have obvious aggregation and size growth phenomenon, so that the dislocation is easy to pass through the particles, and the stress concentration phenomenon is not easy to occur, at the same time, the movement distance of free electrons increases, and reduce the free electrons scattering effect, therefore, the hardness of the alloy decreases, but the electrical conductivity is obviously improved. In addition, at higher aging temperature, η′ phase and η phase can nucleate directly from the solute atom aggregation region, that is, the temperature and time of secondary aging have an important influence on the type and size of precipitated phase, the width of PFZ and the properties of matrix, which will affect the mechanical properties and electrical conductivity of the alloy. In 160 °C two-stage aging process, due to the low temperature, the precipitation power of the second phase in the alloy is insufficient, so the aging holding time is prolonged, and the precipitation quantity of strengthening phase is insufficient, which makes the improvement effect of hardness of the alloy poor, but the improvement of conductivity is obvious. In 180 °C two-stage aging process, the precipitation size of strengthening phase is smaller, the quantity is more uniform, and the hardness and conductivity are better. In the two-stage aging process at 200 °C, due to the higher aging temperature, a large number of η′ and η phases precipitate in the alloy, resulting in the increase of the size of precipitates and a large number of agglomerations of the second phase, resulting in low hardness and good conductivity. To sum up, 470 °C × 1h (60 °C water cooling) + 480 °C × 1h (45 °C water cooling) + 120 °C × 6h (25 °C water cooling) + 180 °C × 2h (25 °C water cooling) is the best heat treatment process with the best comprehensive index of hardness and conductivity.

4. Conclusion

The conclusions obtained through the analysis and research on the hardness and electrical conductivity of the extruded 7075 aluminum alloy are as follows:

1) In the process of heat treatment, solution treatment and aging treatment have insufficient improvement in the hardness of the alloy, but conductivity is improved significantly, and the conductivity and harness are positively linearly correlated.

2) In the two-stage aging process, with the extension of the second stage aging time, the hardness of the alloy increases first, reaches the peak value, and then decreases gradually, while the change trend of conductivity and hardness is opposite, that is, the hardness and conductivity have a very strong negative linear correlation;

3) After heat treatment at 470 °C × 1h (60 °C water cooling) + 480 °C × 1h (45 °C water cooling) + 120 °C × 6h (water cooling) + 180 °C × 2h (water cooling), the second phase precipitates in the matrix are mainly...
spherical, and the grain boundary precipitates tend to be smaller. All these make the hardness of the alloy higher than other heat treatment process, but the above-mentioned microstructural characteristics reduce the degree of free electron scattering. The free path of the electrons is increased, so although the hardness is slightly lower than that of the original sample, the higher conductivity is maintained.

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