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Thermal stability of cold worked microstructure of MP159 alloy①

LU Shi-qiang(鲁世强)[1], HUANG Bai-yun(黄伯云)[2], HE Yue-hui(贺跃辉)[2], TANG Jian-cheng(唐建成)[2]
(1. Department of Materials Science and Engineering,
Nanchang Institute of Aeronautical Technology, Nanchang 330034, China;
2. State Key Laboratory for Powder Metallurgy, Central South University, Changsha 410083, China)

[Abstract] In order to know the longest permissible thermal exposure time during which the cold worked (CW) microstructure would remain unchanged, the effect of the thermal exposure at temperatures ranging from 910 ℃ to 1070 ℃ on the microstructure was investigated for MP159 alloy by optical microscopy and transmission electron microscopy (TEM). Such a study can provide guidance for determining reasonable hot forging parameters of fasteners. The results indicate that the intersecting network of fine platelets in CW microstructure are thermal stable when thermal exposure temperature does not exceed 920 ℃. When thermal exposure temperature exceed 920 ℃, the intersecting network of fine platelets will dissolve, but the thermal exposure temperature has the longest permissible thermal exposure time during which the intersecting network of fine platelets will not dissolve.

[Key words] thermal stability; cold work; MP159 alloy; exposure; intesecting network
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1 INTRODUCTION

MP159 alloy possesses the unique combination of ultra-high strength, ductility and corrosion resistance[1]. It is different from most of nickel-based and cobalt-based alloys, many of which derive their strength from the precipitation of gamma prime or from solid solution effects, whereas MP159 alloy derive its strength from both cold working and aging[2,3]. Solution treatment of MP159 alloy produces a single phase FCC structure, whose tensile strength is about 850 MPa. The followed cold drawing with 48% reduction in cross-section area increases the tensile strength to about 1.585 GPa, and the last aging further increases the tensile strength to about 1.895 GPa.

MP159 is at present widely used for critical fasteners which demand a combination of high strength and high resistance to corrosion[4,5]. Notice that the material is already strengthened by cold working prior to bolt fabrication, and the heads of bolts must be formed by local hot forging. In order to meet the requirement of the bolt application performance, the headed blanks must be free from recrystallization in the areas other than the head. From an easy forming point of view, somewhat higher forging temperature and somewhat longer heating time are expected. However, in order to prevent the cold deformed microstructure in the shank adjacent to the head from recrystallicatioin due to heat conduction, namely, prevent the intersecting network of fine platelets in CW microstructure from dissolution, the forging temperature is expected to be as low as possible, and the heating time as short as possible. In order to reach a balance between them, it is essential to investigate the thermal stability of the intersecting network of fine platelets in CW microstructure at different temperatures, which can provide a guidance for determining reasonable hot forging parameters.

2 EXPERIMENTAL

The original materials used in the present investigation and the sample preparation methods for the optical metallography and the TEM are the same as those stated in Ref. [6].

Various thermal exposure tests were conducted in a high temperature furnace with silicon carbide heating elements. The furnace was heated to the selected temperature and held for 3 h to obtain a uniform temperature in the chamber of the furnace, then sample was put into the chamber for selected time followed by air-cooling (AC) or water-cooling (WC). In the commercial hot forging process of the heads of bolts, induction heating is generally used. The microstructural changes in the shank adjacent to bolt head due to heat conduction are approximately equivalent to those in the samples exposed in high temperature chamber.

3 RESULTS AND ANALYSES

The Rockwell hardness of MP159 in solution heat treated (ST) and CW conditions are about HRC8 and HRC44, respectively (the values lower

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than HRC20 only for reference). The optical micrographs in the ST and CW conditions and the transmission electron micrgraph in the CW condition have been given in Ref. [6]. These micrographs impart that the ST microstructure consists of equiaxed FCC grains with a number of annealing twins, whereas the CW microstructure contains an intersecting network of very fine platelets in each FCC grain. In addition, rather high dislocation density and dislocation tangles between thin platelets can be seen in CW condition. This intersecting network has been identified to be deformation twins[7~9].

The optical microstructures of the CW samples exposed to 910 ℃ for 30, 60 and 90 min have been examined. It is found that the deformed structure prior to thermal exposure remains unchanged. Fig. 1(a) shows the optical micrograph of the sample exposed to 910 ℃ for 1 h, no evidence of recrystallization can be seen. However, in the CW sample exposed to 920 ℃ for 2, 15, 30, 60 and 90 min, the development of the recrystallization can be clearly observed by optical microscopy. Figs. 1(b) and (c) show optical micrographs of the samples exposed to 920 ℃ for 2 min and 30 min respectively. Fig. 1(b) indicates that the recrystallization has already begun after only 2 min at 920 ℃ and the nucleation of recrystallization firstly occurs at the previous FCC grain boundaries. The volume fraction of recrystallized grains increases with time, but a complete recrystallization does not take place up to 1.5 h at 920 ℃. Whereas for the sample exposed to 930 ℃ for only 30 min, a full recrystallization has taken place, as shown in Fig. 1(d). It means that the static recrystallization temperature of CW MP159 is 920 ℃. In the recrystallized area, the intersecting network of fine platelets has been dissolved, which will result in the decreasing of the strength.

When the head of bolt is formed by hot forging, the locally heating temperature generally exceeds 920 ℃ in order to facilitate the forming of the head of the bolt. However, the fastener application performance demands that the shank of the bolt must be free from recrystallization in order to retain the prior cold work strengthening effect. If the hot forging process is not reasonably controlled, recrystallization may occur in the shank adjacent to the bolt head because of heat conduction. The following short-time thermal exposure at elevated temperature are to simulate the thermal exposure situation of the shank near bolt head with a view toward knowing the longest permissible thermal exposure time during which no recrystallization would occur. These data are useful for determining reasonable hot forging parameters.

According to the range of the forging temperatures determined by Lu et al[10,11], the thermal exposure temperatures are selected at the

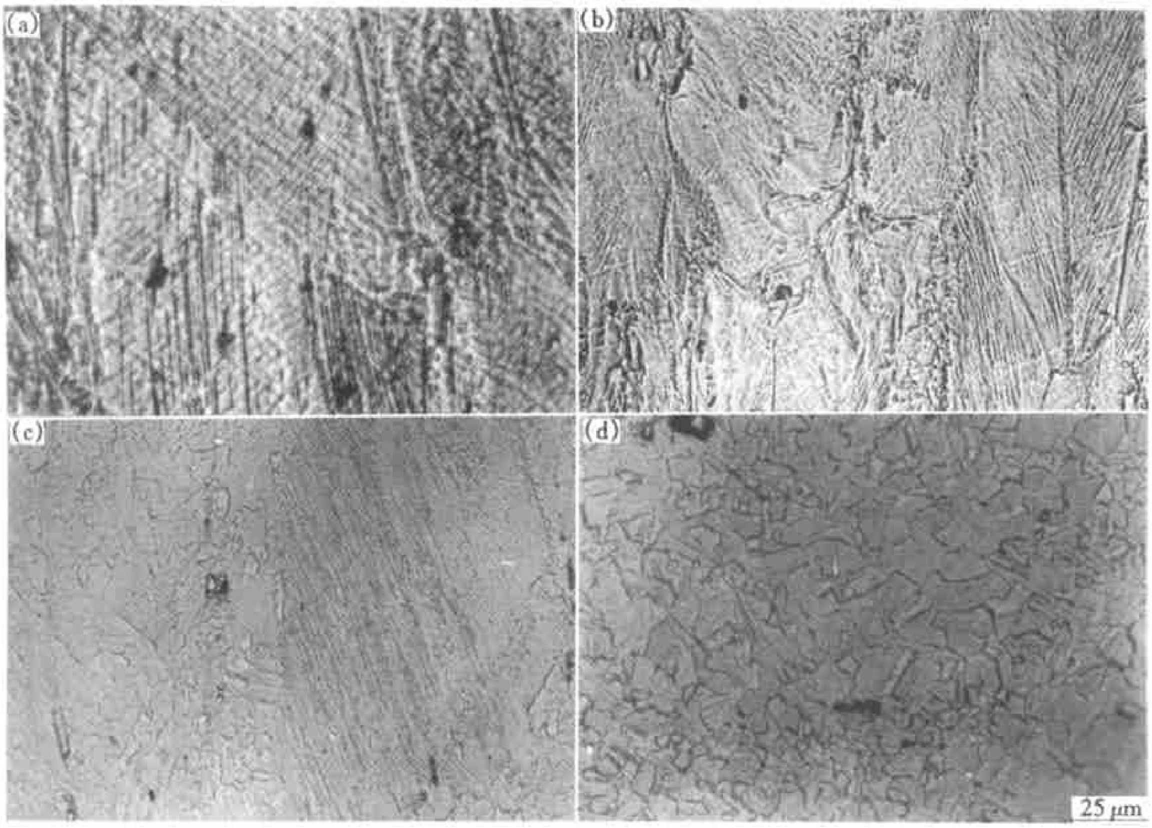

Fig. 1 Microstructures of CW samples after thermal exposure at different temperatures for different durations
(a) —910 ℃ for 1 h; (b) —920 ℃ for 2 min; (c) —920 ℃ for 30 min; (d) —930 ℃ for 30 min

Table 1 Rockwell hardness of thermally exposed samples after differemt modes

Temperature/ ℃, Time/ s	Rockwell hardness (HRC)	
	AC	WC
920, 90	49.8	48.3
920, 90	44.2	43.4
920, 180	38.2	37.1
920, 360	29.8	28.8
950, 90	48.7	47.3
950, 150	29.6	26.5
950, 210	26.8	23.8
950, 300	25.1	23.6
980, 90	43.2	42.3
980, 120	28.1	26.2
980, 150	26.6	25.4
980, 180	25.1	24.3
1010, 40	49.0	47.6
1010, 60	49.5	48.2
1010, 90	27.9	26.3
1010, 150	24.3	23.2
1040, 20	48.6	47.6
1040, 40	50.1	48.8
1040, 70	30.5	27.2
1040, 120	23.4	18.3
1070, 20	49.2	48.8
1070, 40	34.6	33.2
1070, 90	24.3	23.4
1070, 120	17.9	15.0

range of 920~ 1070 ℃. The Rockwell hardness examination of all samples exposed to different temperatures for different times is listed in Table 1. The microstructure of all thermally exposed samples has been observed by optical microscope. The examination of both Rockwell hardness and microstructure reveals that the changing of the microstructure coincides well with the changing of Rockwell hardness, i. e., the larger the volume fraction and/ or the size of the recrystallized grains, the lower the Rockwell hardness. A relationship curve between the Rockwell hardness and the volume fraction of the recrystallized grains is shown in Fig. 2. Generally, when Rockwell hardness is above the value of HRC44, no evidence of recrystallization can be seen, whereas when Rockwell hardness is below the value of HRC28, recrystallization has already finished and new grains begin to grow. On the basis of the examination of Rockwell hardness and optical microstructure, the longest permissible thermal exposure time during which the intersecting network of fine platelets will not dissolve at different temperatures is approximately as follows: 920 ℃, 100s; 950 ℃, 90s; 980 ℃, 70s; 1010 ℃, 60s; 1040 ℃, 40s; 1070 ℃, 20s. It means that when the head of the bolt is formed at the above temperatures, the hot forging process must be finished within the corresponding permissible time, otherwise the recrystallization may occur in the shank near the head of the bolt, resulting in the dissolution of the intersecting network of the platelets.

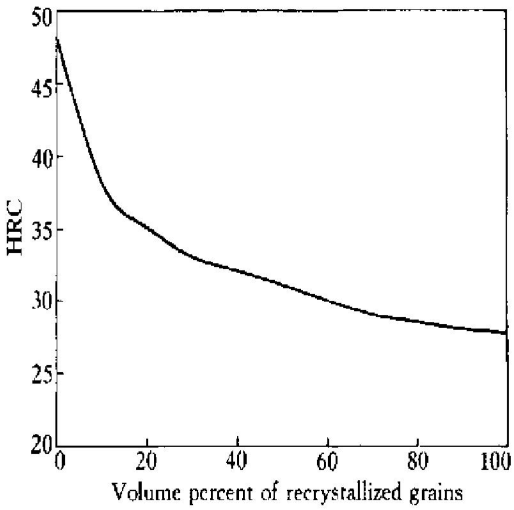

Fig. 2 Relationship between Rockwell hardness and volume fraction of recrystallized grains

In addition, it is strangely seen that although no microstructural changes can be seen by optical examination in the thermally exposed samples in which no recrystallization occurs and in the CW sample prior to thermal exposure, the former has higher Rockwell hardness than that of the later (the hardness of CW sample is about HRC44). Electron diffraction study, as shown in Fig. 3, revealed that there exists the superlattice of γ' ordered phase in these thermally exposed samples. It is this precipitate that resulted in the increase in Rockwell hardness. Obviously, the precipitate forms during the cooling process, which indicates that the formation of the cubic ordered solid solution is very rapid. Such a rapid formation coincides with the observation in MP35N by Drapier et al[12] that age hardening reaches its maximum value after only 15 min.

Another strange phenomenon is that, for the same thermal exposure, the sample cooled by air has somewhat higher hardness than the one cooled by water (see Table 1), although their optical microstructure are identical. It may be associate with the precipitation of γ' phase. Since the samples by AC have more time for the formation of the precipitate than those by WC in the aging temperature range (about 660 ℃), the former could

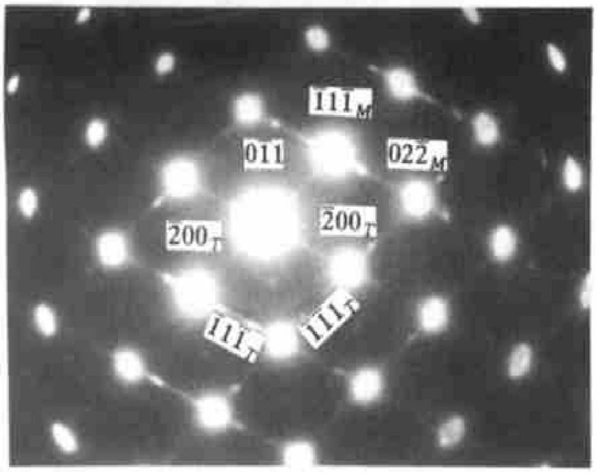

Fig. 3 Selected-area electron diffraction pattern of [011] matrix direction in CW sample exposed to 1 040 ℃ for 40 s and followed by AC

produce more precipitate than the later, resulting in the hardness of the former is somewhat higher than the later.

4 CONCLUSIONS

1) The intersecting network of fine platelets is thermal stable when CW MP159 alloy is thermally exposed at less than 920 ℃.

2) In order to prevent the CW MP159 from recrystallization, i.e., prevent intersecting network of fine platelets in the CW microstructure from dissolution, the longest permissible thermal exposure time at different temperatures is approximately as follows: 920 ℃, 100 s; 950 ℃, 90 s; 980 ℃, 70 s; 1 010 ℃, 60 s; 1 040 ℃, 40 s or 1 070 ℃, 20 s, respectively.

3) The ordered γ' phase is precipitated during cooling process of the thermally exposed samples in which no recrystallization occurs, resulting in the increase of the hardness.

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