# EFFECT OF RESIDUAL STRAIN ON ALPHA CR PRECIPITATION IN ALLOY 718 FASTENERS

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#### Abstract

Alloy 718 is typically used as a fastener material where high strength, corrosion resistance, and elevated temperature service are critical requirements. At elevated temperatures, the precipitation of alpha Cr phase in grain boundaries of alloy 718 can markedly decrease its impact properties. Increased amounts of residual strain in the material dramatically increase alpha Cr precipitation and growth. 1517Mpa (220 ksi) bolts, which are made from 20% cold worked bar, were found to have more alpha Cr than 1241Mpa (180 ksi) bolts both before and after high temperature exposure. Experiments on the 20% cold worked bar revealed that the alpha Cr precipitated during the 718°C part of the direct aging cycle and that a solution treatment of 982°C did not decrease the precipitation. When alloy 718 bolts were exposed at 538°C for 500 hours, severe alpha Cr precipitation occurred adjacent to the cold rolled bolt threads, where alpha Cr was found in a fine network of either grain or subgrain boundaries.

#### Introduction

The versatility of alloy 718 is evidenced by its widespread use in castings and forgings employed by the aerospace, gas turbine, and chemical industries. Alloy 718 is currently used as a fastener material where corrosion resistance and strength are prime requirements. Roach describes the current fastener materials and the factors used for selection of alloy 718 as a fastener material (1). Alloy 718 fasteners are used for applications where minimum tensile strength requirements are 1241 MPa (180 ksi) and 1517 MPa (220 ksi). The 1241 MPa tensile strengths are achieved by standard solution and aging techniques, while the 1517 MPa strengths are obtained by direct aging after approximately 20% cold work. Direct age parts do not undergo a solutioning step and typical aging cycle temperatures are 718°C (1325°F) followed by 621°C (1150°F). As the amount of cold work increases, the ultimate tensile strength increases, and as shown by Roach (1), alloy 718 fasteners experience a severe drop in ductility below a critical level as the ultimate tensile strength exceeds 1655 MPa (240 ksi).

In long time studies of alloy 718 in a temperature range of  $593^{\circ}$ C ( $1100^{\circ}$ F) to  $760^{\circ}$ C ( $1400^{\circ}$ F), it was found that alpha Cr phase forms associated with the delta phase. At  $593^{\circ}$ C, the alpha Cr phase appears without obvious changes in either the  $\gamma''/\gamma'$  precipitation or the strength properties, but the impact properties are lowered(2). Recent studies of direct aged alloy 718 forgings has shown that the residual strain in the forgings enhance the formation of the alpha Cr(3). In addition, it has been shown that cold working alloy 718 greatly increases the kinetics of alpha Cr growth(4). It is believed that the precipitation and growth of the alpha Cr phase in conjunction with the delta phase in severely worked alloy 718 grain boundaries degrades ductility and impact resistance and is the focus of this study.

## **Experimental Procedure**

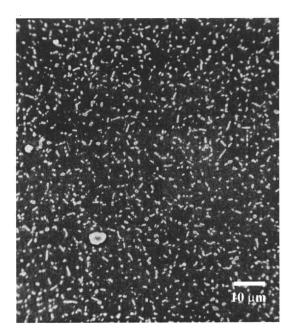
The sample preparation techniques used for this alloy 718 bolt study were the same as described previously to detect the alpha Cr phase(2-4). The alpha Cr phase can be etched out by an electrolytic polish followed by an electrolytic etch in a chromate based solution. This etch preferentially etches chromium rich phases. Electropolishing alone will reveal the alpha Cr in relief, but the contrast is low and it can be difficult to distinguish the alpha Cr and the delta phase. X-ray diffraction on extracted residues was used to verify that the etched out phase is alpha Cr.

A large number of alloy 718 bolts have been structurally evaluated to understand their mechanical performance. Bolt microstructure was examined in the as-received condition and after exposure at 538°C (1000°F), 649°C (1200°F) and 677°C (1250°F) for extended times. The tendency to form the alpha Cr in such tests is a good indicator of the longer time behavior of alloy 718. Bolts with minimum tensile strengths of 1241 MPa (180 ksi) and 1517 MPa (220 ksi) were studied. All bolts originated for a single bolt supplier. A sample of 20% cold work bar was also supplied by the manufacturer.

#### Results

#### As-Received Microstructures

It was found that the alloy 718 used for bolts exhibited large variations in grain size and in the amount and shape of the delta phase. Figure 1 shows two examples of delta phase morphology present in bolts. The variations in microstructure and grain size can be related to the processing prior to the bolt manufacture. Several bolts were examined for alpha Cr in the as-received condition. This was done to provide a starting microstructure and it was expected that no alpha



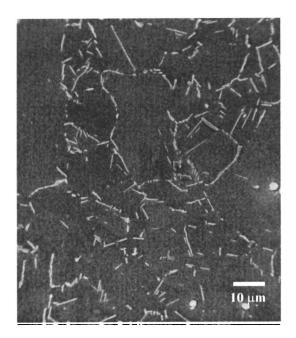


Figure 1. Examples of cross-sectioned bolts showing the variable starting microstructures.

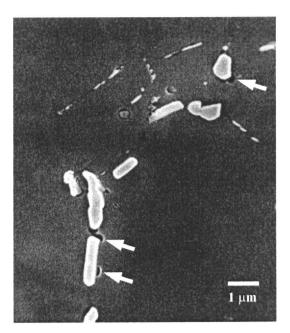


Figure 2. As-received bolt microstructure showing the presence of alpha Cr (arrows mark the etched out holes).

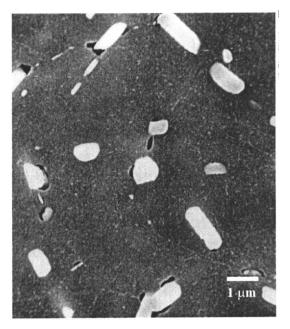


Figure 3. 1241 MPa bolt center after a 649°C exposure for 500 hours.

Cr would be found prior to exposure. However, alpha Cr was found throughout all 1517 MPa and some 1241 MPa bolts and was associated with the delta phase. Figure 2 shows an amount of alpha Cr typical of most bolts, although some bolts were found to contain significantly greater amounts of alpha Cr. No relationship between delta morphology and the amount of alpha Cr in the as received condition was found.

#### **Bolt Centers**

Both 1241 MPa and 1517 MPa bolts were then exposed at elevated temperatures for 500 hours. Figures 3 and 4 show typical microstructures from 1241 MPa and 1517 MPa bolts. Significantly more alpha Cr is found after the thermal exposure, as evidenced by the increase in holes in the

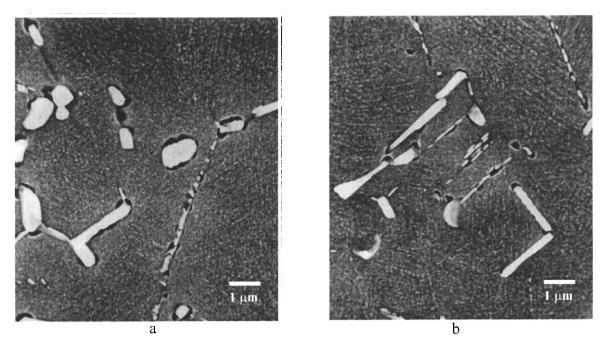


Figure 4. 1517 MPa bolt centers exposed at (a) 677°C for 100 hours and (b) 649°C for 500 hours.

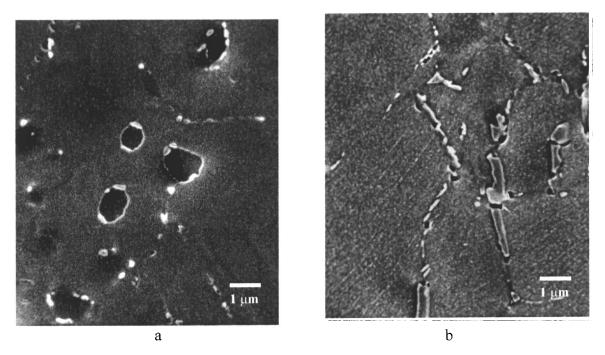


Figure 5. 1517 MPa bolt center exposed at 538°C for 500 hours. (a) electropolished condition (b) electroetched condition.

etched microstructures. It was found that the 20% cold work in the 1517 MPa bolt resulted in increased amounts of alpha Cr, Figure 4. In addition, coarsening of the  $\gamma$ " can be seen in the 1517 MPa bolts. Figure 5 shows a 1517 MPa bolt exposed at 538°C for 500 hours. The alpha Cr can be seen in the electropolished condition and the corresponding holes are visible in the etched condition. The alpha Cr appears to form on both the large delta particles and on the finer grain boundary delta particles.

### 20%CW Bar

A sample of 20% cold worked bar used to make 1517 MPa bolts was obtained to understand the microstructural evolution during processing of a bolt. The bar was examined in the as-cold worked condition, and no alpha Cr was found (Figure 6a). A sample of the bar was then given a typical direct aging treatment of 718°C for 8 hours followed by 621°C for 8 hours. Etching of the sample revealed the presence of alpha Cr. Subsequent tests were run to determine the time/temperature of alpha Cr precipitation in the 20% CW bar. Figure 6b shows a bar sample exposed at 718°C for 8 hours. Alpha Cr precipitation is clearly evident. Samples were also exposed at 704°C for 12 hours and 649°C for 24 hours. The alpha Cr precipitation quickly decreases below 704°C, as no alpha Cr was found during the 649°C exposure.

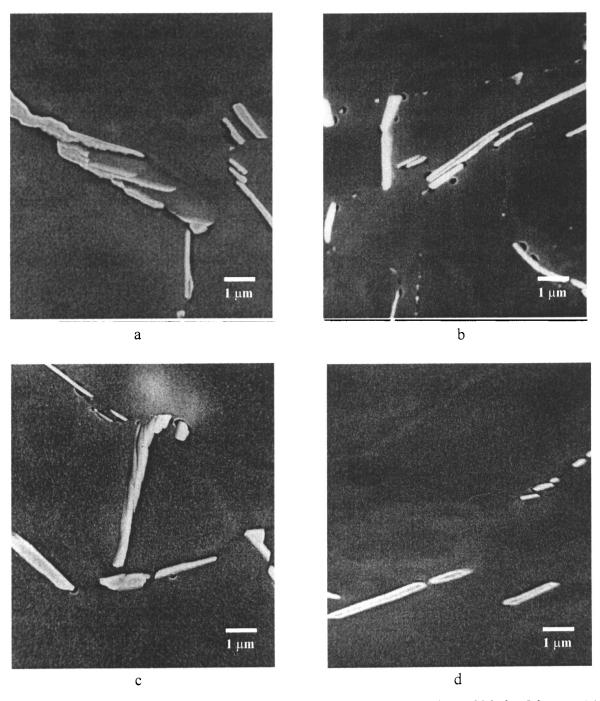


Figure 6. 20% cold worked bar (a) as-received and after exposure (b) 718°C for 8 hours, (c) 704°C for 12 hours and (d) 649°C for 24 hours.

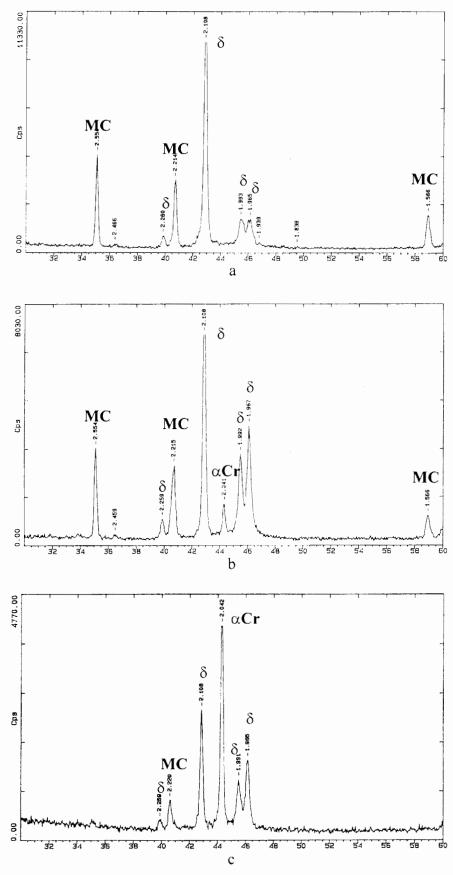


Figure 7. X-ray diffraction spectra of (a) 20% cold worked bar, (b) 20% cold worked bar plus 718°C for 8 hours, and (c) 1517 MPa bolt exposed at 677°C for 100 hours.

In addition to SEM evaluations, phase extractions were carried out on the as-20% cold worked bar, a bar sample aged at 718°C for 8 hours, and a bolt exposed at 677°C for 100 hours. The x-ray analyses of the extracted residues show that the alpha Cr is not present in the as-received 20% cold worked bar, but that alpha Cr is found after 8 hours at 718°C. Much greater amounts of alpha Cr are found after a 677°C for 100hour exposure, Figure 7. The x-ray data confirms the results found via metallography.

Since the residual strain in alloy 718 is thought to accelerate the alpha Cr precipitation process, two 20% cold worked bar samples, one solution treated and one as-received, were compared after aging and exposure. The first sample was solutioned at 982°C for 1 hour and air cooled. Both samples were then aged at 718°C for 8 hours and then exposed at 677°C for 100 hours. No difference in alpha Cr precipitation was found between the two samples, Figure 8. The 1 hour solution treatment was not an effective treatment to prevent alpha Cr. Either the solutioning step did not relieve the residual strain or perhaps the nucleation of alpha Cr occurred on heating or on deformation. No change in delta morphology or grain size was noted during the solution.

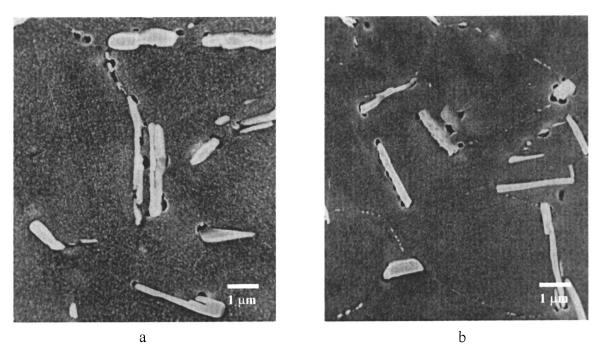


Figure 8. 20% cold worked bar (a) solutioned at 982°C for 1 hour and (b) not solutioned. Both samples were then aged at 718°C for 8 hours and exposed at 677°C for 100 hours.

## **Bolt Threads**

As shown in an earlier section, alpha Cr was found associated with existing delta particles throughout the bolts and no difference in alpha Cr precipitation between the shank and thread regions was found. The alpha Cr formation in the shank portion of the bolt is a result of the original 20% cold work; however, the threads are cold rolled into the bolt after the ages and would have a higher residual strain. To study the alpha Cr formation in the thread areas and the shank portion, a 1241 MPa and a 1517 MPa bolt were exposed for 500 hours at 649°C.

A bimodal distribution on delta was found after exposure in the threaded region of the 1241 MPa bolt. This bimodal distribution consisted of the original larger delta precipitates and finer delta particles that precipitated during the exposure on grain and subgrain boundaries, Figure 9a.

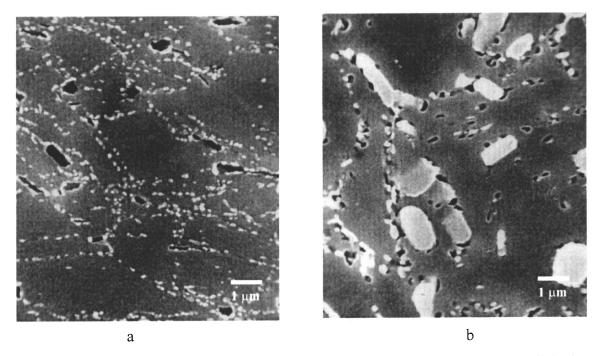


Figure 9. 1241 MPa bolt thread exposed at 649°C for 500 hours. (a) electropolished (b) electroetched.

More significantly, large amounts of alpha Cr formed in this region, far surpassing the amount found in any bolt shank. The sample was re-prepared in the electropolished condition, where the delta appears dark and the alpha Cr white, Figure 9b. A fine distribution of alpha Cr formed along what appears to be network boundaries, perhaps grain or subgrain boundaries. The alpha Cr precipitate appears to be independent of delta phase under this severely deformed condition, although it is possible that fine delta precipitates exist that are not easily revealed with this preparation and are associated with the alpha Cr. A similar response was found in the 1517 MPa bolt, Figure 10, with the newly formed delta precipitates readily visible.

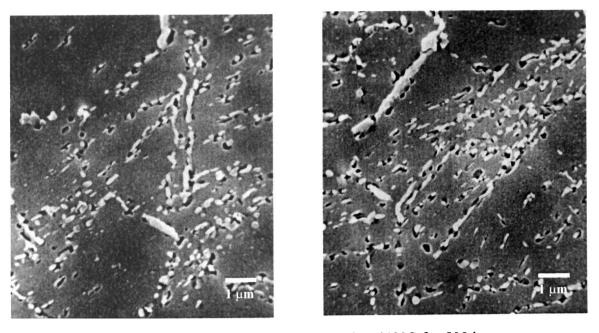


Figure 10. 1517 MPa bolt thread exposed at 649°C for 500 hours.

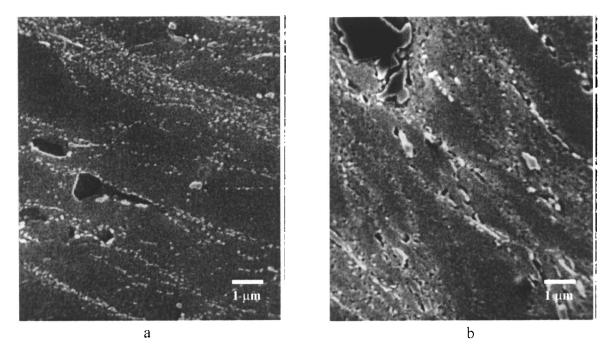


Figure 11. 1517 MPa bolt thread exposed at 538°C for 500 hours (a) electropolished (b) electroetched.

Previous work had shown that alpha Cr precipitation was very sluggish at 593°C in solution and aged material containing low residual strain. The alpha Cr was detected after 25,000 hours at this temperature. To assess the effect high residual deformation on the time and temperature of alpha Cr precipitation, the threaded region of the bolt was examined after exposure at only 538°C for 500 hours. Figure 11a shows very fine alpha Cr precipitates clearly visible in the electropolished condition. The alpha Cr precipitates tend to form in aligned bands with many more nucleated particles than in the 649°C exposure. Very fine delta precipitates were also noted in the etched condition, Figures 11b.

#### Discussion

The amount of alpha Cr is determined by the amount of residual strain. 1517 MPa bolts made from the cold worked bar had greater amounts of alpha Cr than the 1241 MPa bolts. The alpha Cr is first nucleated in the 8 hour age at 718°C and continues to grow in the rest of the age cycle and during high temperature exposures. In addition, the threaded areas form greater amounts of alpha Cr than the bolt center during subsequent exposures due to the added strain from cold rolling in the threads.

The initial structure of bolt material reflects the processing of the material prior to bolt manufacture. Large grain bolts will be more susceptible to continuous grain boundary delta and alpha-Cr formation and thus more susceptible to embrittlement. The variations in delta morphology (discrete, spheroidal precipitates vs. plate-like, acicular precipitates) found in the bolt samples may play a role in the alpha Cr precipitation and material degradation, but no correlation between the different delta morphologies and alpha Cr precipitation was found.

The alpha Cr phase found in bolts is similar to that found in other wrought 718 which had been exposed at 593°C to 760°C for long times. In those studies, when copious amounts of alpha Cr formed, the mechanical properties were downgraded. Since alpha Cr has been associated with a decrease in some mechanical properties, it is expected that the extensive precipitation in the threaded region would severely degrade the mechanical properties in the area immediately

adjacent to the threads. Large diameter bolts would be less affected by this precipitation but as the bolt diameter is decreased, the volume fraction of the affected material will increase and degrade the overall mechanical properties of the bolt.

It was interesting that the solution treatment on the 20% cold worked bar did not decrease the amount of alpha Cr formed after aging and exposure. It had been found that solution and aged forgings formed less alpha Cr than direct aged forgings (3) and it was presumed that this was due to the solution treatment. While the single test on the cold work bar does not disprove this presumption, it may be possible that some residual strain remains in the material whenever it is deformed in the presence of delta. This residual strain could persist through a solution treatment and be responsible for the accelerated alpha Cr precipitation. If this is true, it is advised that solution and aged 1241 MPa bolts are not made from heavily cold worked bar.

#### Conclusions

- 1. Alpha Cr precipitates were found in as-received 1517 MPa (220 ksi) bolts. The combination of the prior cold working of the input material and the direct aging cycle resulted in the precipitation of the alpha Cr.
- 2. Aging of 20% cold worked bar revealed that alpha Cr readily precipitated at 718°C while significantly less was found at lower temperature and longer times. Solutioning the cold worked bar at 982°C did not affect the precipitation of alpha Cr after aging and exposure.
- 3. After long term exposure at elevated temperature, more alpha Cr was found in 1517 MPa (220 ksi) bolts than 1241 MPa (180 ksi) bolts due to increased residual strain.
- 4. Severe alpha Cr precipitation was found in the material adjacent to the bolt threads at temperatures as low as 538°C. Precipitation occurred on a network of what appeared to be grain or subgrain boundaries.

# References

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