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# Temper Developments Using Secondary Ageing

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## Abstract

Secondary ageing may occur if age hardenable aluminium alloys are first underaged at an elevated temperature (eg. 150°C), quenched and then exposed to a lower temperature (eg. 25-65°C). At these lower temperatures, nucleation of fine precipitates occurs that further depletes the microstructure of solute elements and gives rise to additional strengthening. This treatment has been designated the T6I4 temper (I= interrupted) by the authors, and alloys normally develop tensile properties close to those for the respective T6 tempers. If these alloys are then aged again at an elevated temperature (T6I6 temper) further increases in tensile properties are possible (e.g.10-15%), usually with simultaneous increases in fracture toughness. Microstructural changes associated with these improved properties are discussed and examples are given of other tempers that have been developed to meet specific service requirements.

## 1. Introduction

Multi-stage heat treatments provide a means for modifying the size, composition, species, morphology and distribution of precipitate particles in aged aluminium alloys. These changes can result in improvements to mechanical and other properties [eg. 1-6]. An example of a multi-stage heat treatment is the T73 temper in which artificial ageing at one temperature (eg. 100°C) is followed by a second treatment at a higher temperature (eg. 160°C). The T73 temper increases the stress corrosion resistance of 7000 (Al-Zn-Mg-Cu) series alloys by modifying the microstructure, although some sacrifice in tensile properties occurs compared with the single stage, T6 temper [1,2]. Another treatment is to naturally age at room temperature after quenching and before artificial ageing. Such a delay period, which may be unavoidable during normal industrial processing, can increase strengthening of some 7000 series alloys [1] and casting alloys such as 356 and 357, whereas tensile properties are reduced in some 6000 series alloys [3]. In other alloys, a duplex treatment of this kind causes retrogression in which fine clusters or precipitates formed at low temperatures re-dissolve on elevated temperature ageing.

For many years, it was accepted that once an aluminium alloy was artificially aged, the microstructures and mechanical properties remained stable when the alloy was subsequently held for extended periods at a significantly lower temperature.

Then it was recognized that secondary precipitation may occur which, in some alloys, can result in a loss of ductility and fracture toughness [5-7].

Now it has been shown that secondary precipitation may be utilized to enhance mechanical properties through the use of so-called interrupted ageing procedures [8-11].

One such process, which involves interrupting a normal T6 temper with a period when the alloys are held at a lower temperature (eg. 65°C), has resulted in significant improvements in tensile properties and fracture toughness [8,9]. A series of different interrupted ageing tempers have now been developed which are summarized in Figure 1 and Table 1. The purpose of the present paper therefore, is to report some of the effects of these various interrupted ageing treatments on the mechanical properties of selected aluminium alloys.

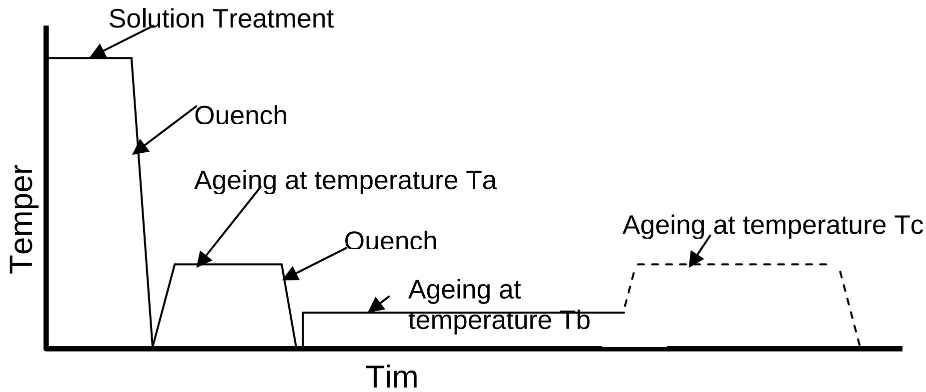


Figure 1: Schematic representation of interrupted ageing tempers.

Table 1: Interrupted ageing temper variants.

| Temper | Process |
|---|---|
| T6I6 | Solution treat, quench, underage at (Ta), quench, age at 25°C-65°C (Tb), re-age at artificial ageing temperature (Tc), where Tc≤Ta. |
| T6I76 | Solution treat, quench, underage at Ta, quench, age at 25°C-65°C (Tb), re-age at artificial ageing temperature (Tc), where Tc>Ta. |
| T8I6 | Solution treat, quench, cold work, underage at Ta, quench, age at 25°C-65°C (Tb), re-age at artificial ageing temperature (Tc), where Tc≤Ta. |
| T9I6 | Solution treat, quench, underage at Ta, cold work, age at 25°C-65°C (Tb), re-age at artificial ageing temperature (Tc), where Tc≤Ta. |
| T6I4 | Solution treat, quench, underage at (Ta), quench, age at 25°C-65°C (Tb) |
| T6I7 | Solution treat, quench, underage at (Ta), cool slowly (furnace cool or natural cool). |
| T77I4 | Solution treat, quench, age at (Ta1), age at temperature (Ta2), where Ta2>Ta1, quench, age at 25-65°C (Tb) |
| T8I4 | Solution treat, quench, cold work, underage at (Ta), quench, age at 25°C to 65°C (Tb). |
| T9I4 | Solution treat, quench, underage at (Ta), cold work, age at 25°C-65°C (Tb) |

## 2. Experimental

The alloys were solution treated in air at appropriate temperatures and quenched into water at 20°C. They were then artificially aged in oil at temperatures used for conventional T6 tempers for times needed to reach 50-85% of peak hardness, quenched into a suitable solvent (Solvex D60), and held at 25°C or 65°C.

Where necessary, alloys were then re-aged to achieve peak hardness at, or close to, the first ageing temperature. Where a cold working stage was required, this was achieved by deforming the alloys either 3% for T651 type tempers or 5% for T8 tempers. Vickers hardness testing was conducted using a 10kg load and transmission electron microscopy was conducted using a JEOL 2000 EX electron microscope operating at 200kV.

## 3. Results and Discussion

### 3.1 T6I4 Temper.

The T6I4 temper utilizes secondary precipitation after a short period of underageing at an elevated temperature to achieve, or sometimes to surpass, the properties of the T6 temper, but with notably reduced times of artificial ageing. Figure 2 shows examples of this behaviour for the wrought alloys 7050, 2001, 8090, and the casting alloy 357. In each case, the hardness of the T6I4 material (dashed lines) begins to equal, or to exceed that of the corresponding T6 temper (solid line). Of particular interest are the secondary ageing curves for the Li-containing alloy, 8090. A surprising result is that material aged 2, 4 or 6 hours, initially hardens more rapidly during secondary ageing at 65°C than if ageing was continued at 185°C. After 8 hours at 185°C, the rate of secondary ageing at 65°C is now slower than occurs by single stage artificial ageing at 185°C.

Alloys aged to the T6I4 temper display tensile properties close to, and sometimes exceeding those of the T6 tempers. In most instances, the fracture toughness is also significantly improved, as shown for the casting alloy 357, and the wrought alloys 7050 and 6061 (Table 2). For other alloys, such as the Al-Cu-Mg alloys 2001 and 2214, the fracture toughness is similar to that measured for the T6 temper.

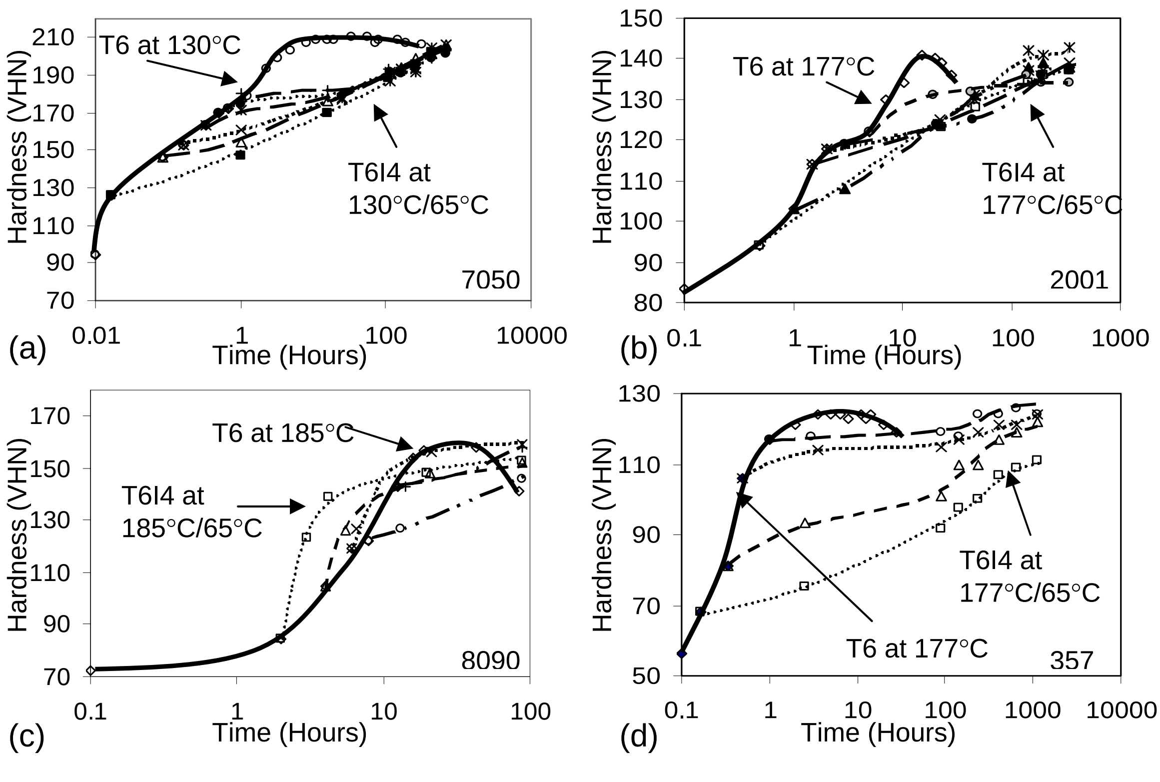


Figure 2: Secondary ageing curves (T6I4) compared with T6 curves for (a) 7050, (b) 2001, (c) 8090 and (d) 357.

Prolonged T6I4 treatments exceeding 35000h were conducted on the alloys 7075 and 7075+0.3wt% Ag, which were aged initially for 0.5h at 130°C, and then held at either 25 or 65°C. For secondary ageing of 7075 at 25°C, the hardness continued to rise for approximately 10000h when it reached 205VHN, slightly exceeding the T6 peak value of 197VHN (Figure 3a). When secondary ageing was conducted at 65°C, the hardness rose to the higher value of 222 VHN.

Ag is known to promote increased hardening in 7000 series alloys aged at elevated temperatures [12] and the peak hardness for 7075+Ag in the T6 temper is higher at 212VHN. In this case, the T6I4 treatment at 25°C also resulted in a peak hardness after 10000h of 205VHN, but this value is lower than the T6 hardness for 7075+Ag. This effect may be attributed to a reduced mobility of vacancies at 25°C due to their preferred interaction with Ag atoms [13]. However, at 65°C, the T6I4 temper resulted in a remarkably high hardness of 240VHN, which suggests that Ag may promote formation of a more potent strengthening phase in 7075.

Table 2: Mechanical properties for alloys in the T6 and T6I4 tempers.

| Alloy and Treatment | 0.2% Proof Stress (MPa) | UTS (MPa) | % Elongation at Failure | S-L Fracture Toughness (MPa√m) |
|---|---|---|---|---|
| 7050 T6 | 546 | 621 | 14 | 37.6 |
| 7050 T6I4 | 527 | 626 | 16 | 52 |
| 2214 T6 | 386 | 446 | 14 | 26.9 |
| 2214 T6I4 | 371 | 453 | 13 | 27.1 |
| 2001 T6 | 265 | 376 | 14 | 56.3 |
| 2001 T6I4 | 260 | 420 | 23 | 56.9 |
| Al-Cu-Mg-Ag T6 | 442 | 481 | 12 | 23.4 |
| Al-Cu-Mg-Ag T6I4 | 443 | 503 | 8 | 28.1 |
| 6111 T6 | 339 | 406 | 13 | ---- |
| 6111 T6I4 | 330 | 411 | 14 | ---- |
| 6061 T6 | 267 | 318 | 13 | 36.8 |
| 6061 T6I4 | 302 | 341 | 16 | 43.2 |
| 357 T6 | 287 | 340 | 7 | 25.5 |
| 357 T6I4 | 280 | 347 | 8 | 35.9 |

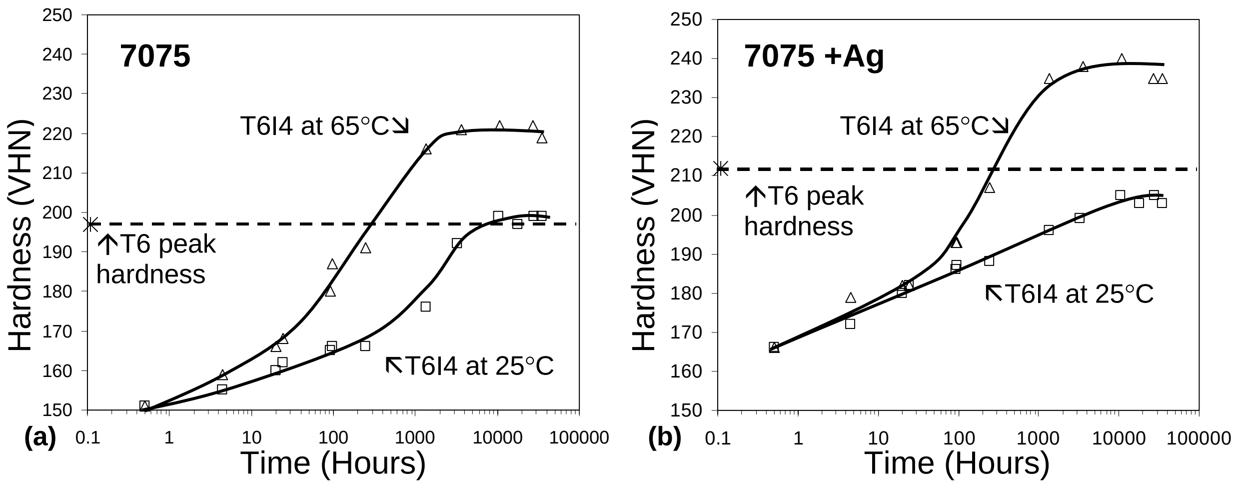


Figure 3: Comparisons of the T6I4 temper at 25°C and 65°C for (a) alloy 7075 and (b) 7075+Ag.

## 3.2 Variants of the T6I4 Temper

### 3.2.1 T8I4 and T9I4 Tempers.

These two tempers incorporate cold work into their processing schedule. For the T8I4 temper, an alloy is cold worked after solution treatment and before commencing ageing whereas the T9I4 schedule involves cold work after preliminary ageing and before secondary ageing. Both exhibit continuing secondary precipitation after each of the treatments. An example of this behaviour is shown in Figure 4 for the 2000 series alloy, 2001, which has a hardness of 83VHN after quenching from the solution treatment temperature. The application of 10% cold work by rolling raised this hardness to 98VHN and, on artificial ageing at 177°C, the peak value (T8 temper) was 140VHN. For the T8I4 temper, the alloy was cold worked 10% after quenching from the solution treatment temperature. It was then aged at 177°C for 0.5h and again quenched. The hardness was then 105VHN, and subsequently increased to 135VHN after 400h at 65°C.

For the T9I4 temper, the solution treated and quenched alloy was aged 1.5h at 177°C, and again quenched, the hardness then being 114VHN. Cold working 10% raised this value to 123VHN, which increased to 143VHN after 400h at 65°C, a value slightly higher than for the T8I4 temper.

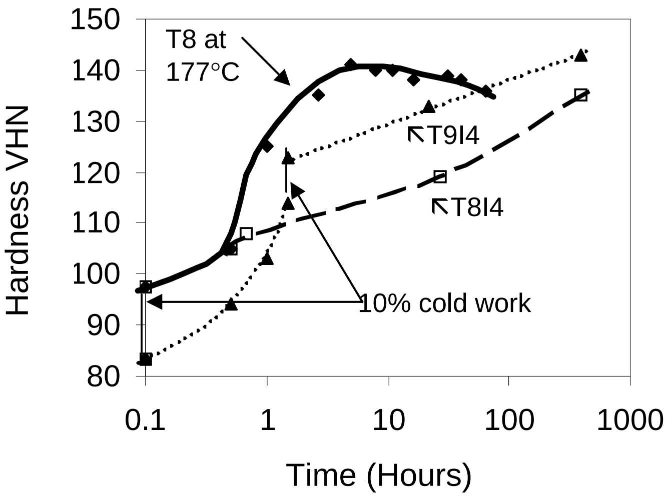


Figure 4: T8I4 and T9I4 hardening curves for the alloy 2001. See text for details.

### 3.2.2 T6I7 Temper

This temper is produced in a manner such that secondary ageing is deliberately slowed, or stopped, by slow cooling rather than quenching from the preliminary ageing temperature. It has been found in an Ag containing 2000 series alloy that this treatment may retain the superior creep resistance produced by underageing prior to creep exposure [14]. This situation is summarized in Figure 5. Figure 5a shows the secondary creep rate for underaged alloy as a function of dwell period after underageing, where creep testing was conducted at 150°C and 300MPa. Here it is seen that after approximately 600h dwell at 25°C, the secondary creep rate deteriorates, eventually equalling that of the T6 condition. Secondary precipitation occurs during this period (Figure 5b), but this is suppressed by slow cooling from the ageing temperature (Figure 5c). This treatment retains the superior creep resistance of the underaged condition [14].

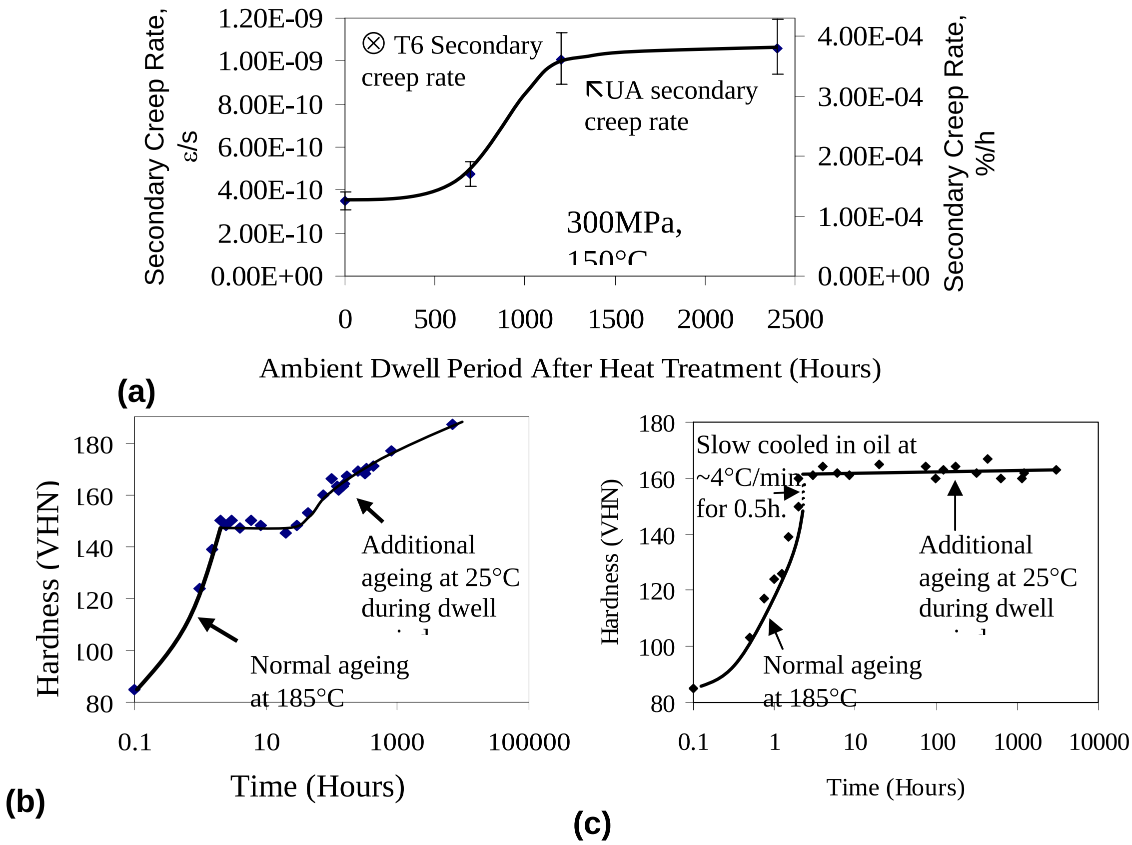


Figure 5: Effect of secondary ageing on creep resistance; (a), underaged material left for varying dwell periods at 25°C showing effect on secondary creep rate; (b), secondary precipitation at 25°C and (c), absence of secondary precipitation following slow cooling from the underageing temperature [14].

### 3.2.3 T77I4 temper

In this instance, the alloy is first artificially aged to peak hardness (T6 temper) and heated to a higher temperature, so that retrogression occurs before it is quenched to room temperature to promote secondary ageing. This produces a microstructure that has previously been assumed to be overaged, through loss of solute to grain boundaries and other solute sinks. Nevertheless, results in Figure 6 show that 7075 alloy responds well to secondary hardening at the low temperature. This complex ageing cycle produces a microstructure containing two dominant precipitate phases; large particles of the $\eta$ phase that were stable enough to be retained during high temperature ageing, interspersed with fine GP zones formed during secondary ageing. In contrast to the T7X tempers commonly used for 7000 series alloys, invoking secondary precipitation does not produce a decrease in mechanical properties of the alloy, with tensile properties for the T77I4 temper being equivalent to those for the T6 temper. This temper has been developed as being potentially suitable for stress corrosion cracking resistance in 7000 series alloys.

Microstructural changes occurring during the T77I4 treatment are not unlike those produced during welding of age hardened alloys.

When these alloys aged to a T6 temper are welded, regions in the parent metal become overaged due to precipitate coarsening and re-solution of solute. These regions that are softened may be partially re-hardened by secondary ageing at or close to ambient temperature if the alloy is quenched immediately after welding.

This effect is illustrated in Figure 7 for the alloy 6061-T6 for which the hardness profile across a weld and the adjacent heat affected zone (HAZ) has been monitored for times up to 6400h. For the sample air cooled after welding (Figure 7a), only the central region of the weld increases in hardness, similar to what would occur for a T4 ageing treatment. In the quenched condition, (Figure 7b), the region that has regressed has partially re-hardened which decreases the width of the HAZ and improves the tensile properties of the weld zone. The effect of this quenching on the tensile properties of the welded alloy is shown in Table 3.

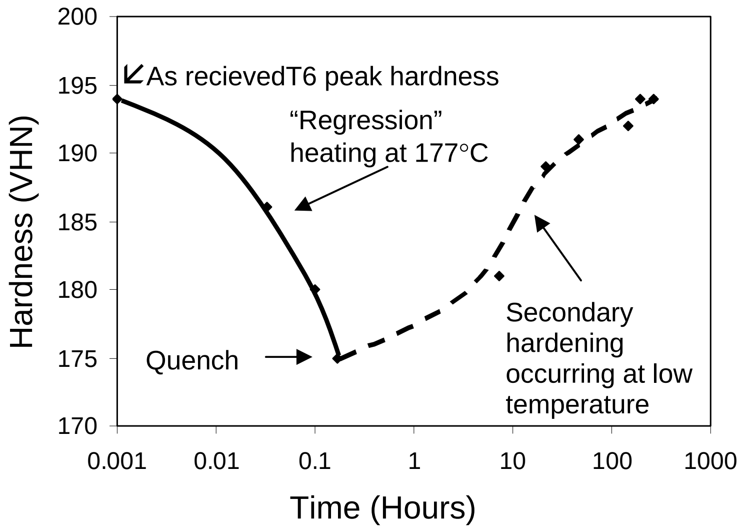


Figure 6: Example of the T77I4 temper for 7075 alloy. T6 aged material is put through a regression treatment, that re-dissolves solute so that secondary precipitation is then able to occur.

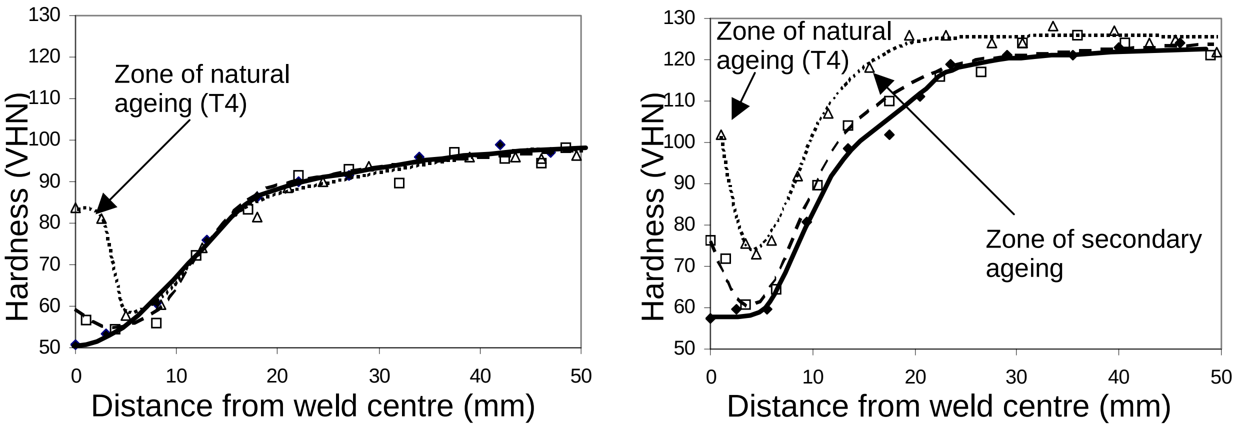


Figure 7: Changes in hardness associated with a weld either a), naturally cooled after welding, or b), quenched into water after welding. Solid line shows hardness immediately after welding, squares show hardness after 66h and triangles hardness after 6400h.

Table 3: Effect of quenching on weld zone properties of alloy 6061 in the T6 condition.

| **Sample** | **0.2% proof stress** | **UTS** | **% Elongation** |
|---|---|---|---|
| Naturally air cooled | 118 MPa | 185MPa | 10% |
| Water quenched | 159MPa | 219MPa | 8% |

### 3.3 T6I6 Temper

The T6I6 temper is completed when an alloy that has undergone a complete or partial T6I4 temper, is then aged again at an elevated temperature for a time to reach peak strength. Typically, average improvements of 10-15% to hardness, yield strength and tensile strength occur during this ageing procedure, with simultaneous improvements to fracture toughness in most alloys. Changes that may occur in microstructure are shown for the Al-Cu-Mg alloy 2014 in Figure 8, together with the respective ageing curves for the T6I6 and T6 conditions. Both the size and dispersion of precipitate phases (θ' and S(S')) have been modified by the T6I6 temper. Moreover, quantitative analysis of these phases has shown that the number of the S(S') precipitates is increased by some 75% as a result of the T6I6 temper.

Tensile property comparisons of a range of alloys aged to the T6I6 and T6 temper are shown in Table 4, and fracture toughness values are shown in Table 5. For the alloy 8090 significant improvements in the fracture toughness of this alloy are produced by a T6I6 temper, whereas this did not occur with the T6I4 temper.

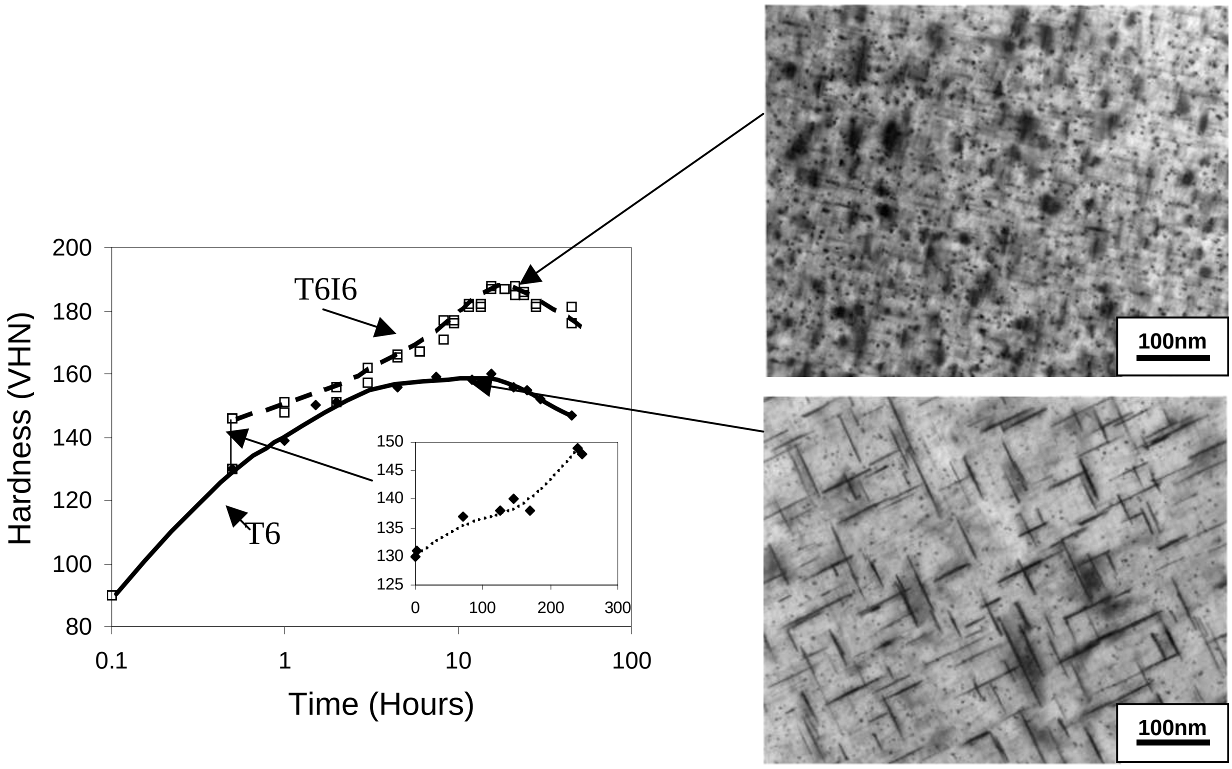


Figure 8: Interrupted ageing hardness curves and microstructures for the Al-Cu-Mg alloy 2014. The dashed line in the main diagram is represented by the inset plot showing secondary hardening (vertical axis) as a function of dwell time at 65°C (horizontal axis). In addition to its refined microstructure, the T6I6 microstructure contains 75% more S phase rods than are present after a T6 temper.

### 3.4 Variants of the T6I6 Temper

#### 3.4.1 T6I76 Temper

This temper has been derived from the T6I6 temper and involves a relatively high final ageing temperature (Table 1). As for the T77I4 temper, the T6I76 treatment was also designed to promote a higher resistance to stress corrosion cracking.

Experiments with the alloy 7050 have shown that the T6I76 treatment causes modifications to the microstructure while retaining tensile properties equivalent to those for the T6 temper. Work is ongoing to characterize the microstructure-property relationships in both the T77I4 and T6I76 tempers, and preliminary work has indicated that corrosion and stress corrosion resistance may be improved in both cases.

### 3.4.2 T8I6 and T9I6

As with the T8I4 and T9I4 tempers mentioned previously, the T8I6 and T9I6 tempers incorporate cold work in their ageing cycles, again either before or after preliminary artificial ageing (Table 1). Tensile properties for the T8I6 temper for the alloys 8090 and Al-4Cu are shown in Table 6. In Table 7, the tensile properties for the alloy 6056 are shown for a wider variety of tempers, some of which include a stage involving 3% cold work. The application of cold work during interrupted ageing tempers may produce additional benefits to the mechanical properties of some alloys, particularly in the T9I6 temper where improvements may be greater than those observed from T6I6 tempers. The effect of cold work in the T8I6 temper however, may moderately diminish the magnitude of improvements noted for the T6I6 temper.

Table 4: Tensile Properties of Alloys Aged by T6 and T6I6 Treatments.

| **Treatment ⇒** | **T6** | | | **T6I6** | | |
|---|---|---|---|---|---|---|
| **Alloy** | **0.2% Proof Stress (MPa)** | **Tensile Strength (MPa)** | **Elongation %** | **0.2% Proof Stress (MPa)** | **Tensile Strength (MPa)** | **Elongation %** |
| **Al-4Cu** | 236 | 325 | 5 | 256 | 358 | 7 |
| **2014** | 414 | 488 | 10 | 436 | 526 | 10 |
| **Al-Cu-Mg-Ag** | 442 | 481 | 12 | 502 | 518 | 7 |
| **6061** | 267 | 318 | 13 | 299 | 340 | 13 |
| **6013** | 339 | 404 | 17 | 380 | 416 | 15 |
| **7050** | 546 | 621 | 14 | 574 | 639 | 14 |
| **7075** | 505 | 570 | 10 | 535 | 633 | 13 |
| **8090** | 349 | 449 | 4 | 391 | 512 | 5 |
| **357** | 287 | 340 | 7 | 341 | 375 | 5 |

Table 5: Fracture Toughness of Alloys Given T6 and T6I6 Treatments (S-L orientation).

| **Alloy** | **T6 MPa√m** | **T6I6 MPa√m** |
|---|---|---|
| Al-Cu-Mg-Ag | 23.4 | 30.3 |
| 2014 | 26.9 | 36.2 |
| 6061❋ | 36.8 | 58.4 |
| 7050 | 37.6 | 41.1 |
| 8090 | 24.2 | 31.0 |
| 357 | 25.5 | 26.0 |

Average results for 3 or more tests on each alloy. ❋ Plane strain conditions not possible for alloy 6061.

Table 6: Effect of Cold Work on Tensile Properties.

| **Treatment ⇒** | **T8 Temper** | | | **T8I6 Temper** | | |
|---|---|---|---|---|---|---|
| **Alloy** | **0.2% Proof Stress (MPa)** | **Tensile Strength (MPa)** | **Elongation %** | **0.2% Proof Stress (MPa)** | **Tensile Strength (MPa)** | **Elongation %** |
| Al-4Cu (5% CW) | 242 | 339 | 7 | 265 | 358 | 7 |
| 8090 (5% CW) | 414 | 495 | 5 | 441 | 518 | 5 |

Table 7: Effect of Cold Work on Tensile Properties for Alloy 6056.

| Temper | 0.2% Proof Stress (MPa) | Tensile Strength (MPa) | Elongation% |
|---|---|---|---|
| T6 | 350 | 417 | 17 |
| T6I6 | 381 | 416 | 16 |
| T651/T8 (3%CW) | 342 | 386 | 16 |
| T8I6 (3%CW) | 363 | 389 | 16 |
| T9I6 (3%CW) | 398 | 433 | 19 |

## 4. Conclusions

Interrupted ageing of aluminium alloys may be applied to a wide range of tempers, producing significantly different results. In addition, microstructures tailored for performance are achievable through utilization of secondary aged materials.

1. The T6I4 temper produces tensile properties close to, and occasionally greater than those for the T6 condition together with enhanced fracture toughness in most alloys.
2. T8I4 and T9I4 tempers also are effective in utilizing secondary precipitation. In these instances, cold work is applied at different stages of the ageing cycles.
3. Slow cooling after the initial artificial ageing treatment (T6I7 temper) impedes secondary precipitation at a lower temperature. This treatment may be used to retain superior creep properties in underaged Al-Cu-Mg-Ag alloys.
4. T77I4 tempers, which include a stage of ageing at a high temperature to re-dissolve some precipitates, produce microstructures not unlike those produced in the HAZ adjacent to welds. Secondary ageing then allows partial re-hardening to occur in these zones which reduces their width and increases their strength. These tempers also have the potential to improve stress corrosion resistance in certain alloys.
5. T6I6 tempers typically display simultaneous improvements in the yield strength, tensile strength, hardness and fracture toughness of most alloys. In some cases (eg. 6061) fracture toughness may be increased by as much as 60%. Alternatively, modified microstructures may be produced by altering the temperature of the final artificial ageing step (eg. T6I76 temper).
6. T8I6 and T9I6 tempers in which cold work is employed at different stages of the ageing cycles prior to final elevated temperature ageing, may also produce improved mechanical properties.
7. Interrupted ageing may change the size, dispersion and ratios of precipitate phases.

## Acknowledgements

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