KERNFORSCHUNGSZENTRUM

KARLSRUHE

November 1970 KFK 1318

Institut für Material- und Festkörperforschung
Projekt Schneller Brüter

Room Temperature Tensile Properties and Microstructures of Steel X8CrNiMoVNb 1613 (4988), Incoloy 800 and Inconel 718 after Ageing at 600 to 800°C.

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Room temperature tensile properties and microstructures of steel X8CrNiMoVNb 1613 (4988), Incoloy 8oo and Inconel 718 after ageing at 6oo to 8oo°C.

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Abstract

The room temperature tensile properties of all three alloys are correlated with the microstructural changes as observed by the electron microscope. Steel 4988 shows some hardening after prolonged ageing at 6oo°C due to the precipitation of a very fine V(CN) phase. At higher temperatures this phase coarsens rapidly and no hardening is observed. Other carbides in this alloy do not contribute significantly to the mechanical properties except for $M_{23}C_6$ on grain boundaries, which is found only at 6oo°C and possibly contributes to the lower ductilities of this alloy after ageing at 6oo°C. Similarly, Incoloy 8oo is hardened by ageing at 6oo°C, probably due to the formation of pre-precipitation zones of Ni_3(TiAl). Inconel 718 is hardened by ageing at 6oo and 7oo°C due to the precipitation of a metastable bct-phase. The formation of NbC films on grain boundaries at 7oo°C, can lead to very low ductilities. It is shown that upon overageing, the transformation from the metastable bct- γ'phase to the stable Ni_3Nb phase occurs with the concomitant dissolution of these NbC films on the grain boundaries, causing an increase in the ductility once again.

Kurzfassung

Die Festigkeitseigenschaften bei Raumtemperatur aller drei Legierungen werden mit den strukturellen Änderungen, wie sie im Elektronenmikroskop beobachtet werden können, in Beziehung gebracht. Stahl vom Typ 4988 zeigt etwas Aushärtung nach längerer Auslagerung bei 6oo°C, die auf Ausscheidungen einer sehr feinen V(CN)-Phase zurückgeführt wird. Bei höheren Temperaturen vergröbert sich diese

Phase sehr rasch, so daß keine Aushärtung mehr beobachtet wird. Andere Karbide in dieser Legierung tragen nicht deutlich zu den mechanischen Eigenschaften bei, außer $M_{23}C_6$ in den Korngrenzen, das aber nur bei 600°C gefunden wird und möglicherweise zu der niederen Dehnung dieser Legierung nach Auslagerung bei 600°C beiträgt. Ebenso härtet Incoloy 800 aus nach einer Alterung bei 600°C, wahrscheinlich aufgrund der Bildung von $Ni_3(TiAl)$-Zonen. Inconel 718 wird nach Alterung bei 600°C und 700°C durch die Ausscheidung einer metastabilen t.r.z.-γ'-Phase ausgehärtet. Die Bildung von NbC-Schichten an den Korngrenzen bei 700°C kann zu sehr niedrigen Dehnungswerten führen. Es wird gezeigt, daß nach Überalterung die Umwandlung der metastabilen t.r.z.-γ'-Phase zur stabilen Ni_3Nb-Phase stattfindet bei gleichzeitiger Auflösung der NbC-Schichten an den Korngrenzen, was wieder eine Zunahme der Dehnung bewirkt.

Introduction

Steel 4988 is quite promising as a potential cladding material in a sodium cooled fast reactor fuelled with UC. One of the deciding factors in the selection of a cladding material for such a reactor is the resistance to undesirable interactions with the fuel which can lead to a premature cladding rupture. The effects of incompatibility interactions with UC on the room temperature tensile properties is, therefore, being studied at temperatures between 600° and 800°C on the steel 4988, Incoloy 800 and Inconel 718. The latter two alloys are not very strong candidates for application in a reactor of the above type but were included in this investigation for comparison to the more ideally suited steel 4988.
A preliminary investigation was, therefore, carried out on the annealing behaviour of these alloys under vacuum at the same temperatures and this behaviour was correlated with the room temperature tensile properties. This report is concerned with these latter results viz. the microstructural behaviour and tensile properties of these alloys annealed in vacuum. In particular, attention was paid to the different microstructural phases in the alloys and the changes these undergo during prolonged ageing.
An attempt was made in each case to correlate these changes with the corresponding changes in mechanical properties. These results will be used later in correlation with the compatibility investigations to study the microstructural interactions between UC and these alloys and the effects these have on the mechanical properties.

Experimental Techniques

The steels were received in sheet-form with nominal thickness 1 mm and from these, Vickers-Armstrong tensile test specimens (type Mol) with a gauge length of 25 mm, were die-pressed. The composition of each steel is given in Table I.

These tensile test specimens were then given the following initial heat treatments: Steel 4988 was solution treated and then age-hardened at 750°C for 3 hours, one batch of the Incoloy 800 was annealed and another was solution treated and finally the Inconel 718 was solution treated. The exact heat treatments and resulting grain sizes are given in Table II. The specimens were then given a light electro-polish to remove the oxide film which was formed during water quenching. The electrolyte employed was a 60 % H_3PO_4, 20 % H_2SO_4 and 20 % H_2O solution at room temperature. After electro-polishing the specimens were sealed in silica capsules under vacuum and then annealed at 600, 700 and 800°C for times of 100, 300, 700 and 1100 hours. The Incoloy 800 specimens in the solution treated condition were annealed only at 700 and 800°C. After annealing the room temperature tensile properties were determined on an Instron tensile testing machine at a strain rate of 1.3×10^{-3}/sec. The 0,2 % yield strength, the ultimate tensile strength and the elongation were determined from two seperate specimens for each final heat treatment and the average value of the two used in the final evaluation.

After completion of the tensile tests one half of the specimen was mounted and shadowed carbon extraction replicas were prepared by the procedure described elsewhere [1]. For the initial etch a solution made up from 33 ml methanol, 17 ml HCl and lo-2o drops H_2O_2 was used for all the steels. As discussed previously, the specimens were lightly overetched and thoroughly washed to eliminate any sectioned particles. The specimens were then shadowed with a 9o % Pt-lo % Ir alloy at an angle of 45° and finally a relatively heavy film of carbon was evaporated onto the surface. The carbon films were finally extracted from the specimens electrolytically by a lo% HCl-methanol solution and examined on a Zeiss electron microscope at 6o KV. To identify the precipitates, selected area diffraction patterns were made from the microstructures and calibrated against ring patterns made from a gold-shadowed replica. In the case of Inconel 718, where some large precipitates were present, electron microprobe analysis was also used to identify these precipitates by their composition. In the same alloy thin foil electron microscopy was used in one instance to identify some extremely fine precipitates which could not be resolved by the technique of carbon extraction replicas.

Steel 4988

Results

The room temperature tensile properties of steel 4988 during annealing at 6oo to 8oo°C are shown in fig. 1 while in figs. 2 and 3 some of the corresponding microstructures are shown. The yield and tensile strengths increased slightly with a corresponding decrease in ductility, during prolonged annealing at 6oo°C. At 7oo°C the yield strength and ductility decreased slightly whereas the tensile strength was little affected by annealing while at 8oo°C a fairly rapid drop in strength and an increase in ductility had occurred after only loo hours.

The microstructure of the steel after the initial ageing treatment (3 hours at 750°C) is shown in fig. 2 (a). The structure consists of relatively evenly spaced particles in the matrix while the grain boundaries are entirely free from precipitates. This matrix precipitate was identified as a carbon-rich Nb(CN) precipitate with a best fit for a lattice parameter of 4.44 Å. In addition to the precipitates shown in fig. 2 (a), a few isolated and very large precipitates were also present after the initial heat treatment. These are not shown in fig. 2 (a) but can be seen in fig. 2 (b). Due to their thickness, satisfactory selected area diffraction patterns could not be obtained from these precipitates but, from the literature [2], these could possibly consist of a coarse Nb(CN) precipitate or the Laves phase $NbFe_2$, both of which are usually present in the as-cast structure and do not dissolve easily during the later solution treatment.

During annealing at 600°C this structure does not change noticeably up to 100 hours but after 300 hours some marked changes have already occurred as shown in figs. 2 (b) and (c). The smaller Nb(CN) precipitates in the matrix are still unchanged but pronounced nucleation of $M_{23}C_6$ has taken place on the grain boundaries and in the matrix a very fine precipitate has started to appear in the background. Upon further annealing to 1100 hours at 600°C the density of the fine precipitates has increased and the amount of $M_{23}C_6$ on the grain boundaries has also increased slightly, as shown in fig. 2 (d). The very fine precipitates could be indexed as nitrogen-rich vanadium carbo-nitrides (VCN) with a best fit for a lattice parameter of 4.14 Å. At a higher magnification these V(CN) precipitates appear thread-like (see fig. 2 (c)) and it may be possible that nucleation had occurred on dislocations, causing some strengthening as shown by the tensile properties of fig. 1. The continuous increase in tensile and yield strength with prolonged ageing time at 600°C would also indicate that fresh nucleation of this V(CN) is still occurring even after long ageing times and that coarsening or over-ageing of this precipitate is still insignificant at 600°C.

After 100 hours at 700°C (fig. 3 (a)) the density of the V(CN) seems even greater than at 600°C and some grain boundaries (especially the twin grain boundaries) are free from $M_{23}C_6$ precipitates. After 1100 hours at 700°C it is apparent that the V(CN) has coarsened considerably as shown in fig. 3 (b). The structures at 800°C are very similar to those at 700°C and are shown in figs. 3 (c) and (d). The volume fraction of the V(CN) appears lower at 800°C than at 700°C but this may be due to a much coarser dispersion. It is interesting to note, however, that at 700°C and 800°C the V(CN) precipitates have coarsened into the form of small rods and spheres and are not thread-like any more as at 600°C. This causes no increase in strength at 700° and even a decrease in strength at 800°C. This decrease in strength at 800°C would also indicate that this strengthening precipitate had already been present after the initial ageing treatment of 3 hours at 750°C (fig. 2 (a)) but probably remained undetected by the technique of carbon extraction replicas. This technique has a lower limit of resolution of only about 20 to 50 Å, depending on the thickness of the carbon film. From fig 3 (d) it is also clear that at 800°C no $M_{23}C_6$ precipitates exist on the grain boundaries any more.

On the whole the appearance of the isolated large precipitates and also of the smaller Nb(CN) in the matrix seemed to remain unchanged by temperature or by prolonged ageing and no coarsening of the Nb(CN) is apparent even after 1100 hours at 800°C.

Discussion

In general the strength properties of this alloy can be correlated quite well with the microstructural observations. The Nb(CN) precipitate in the matrix seems to contribute little to the strengthening of this alloy while at 600°C the very fine V(CN) causes some strengthening even after 1100 hours at 600°C. At 700°C and 800°C this precipitate coarsens relatively quickly, losing most of its effectiveness as a strengthening precipitate. Bungardt and Lennartz [2] have found, in a very similar alloy, two precipitates which were identified by X-ray analysis as a niobium-rich (NbV)(CN) precipitate with a lattice parameter of 4.34 Å and a vanadium-rich (NbV)(CN) precipitate with a lattice parameter of 4.28 Å. If the lattice parameters of 4.44 Å for the Nb(CN) precipitate and 4.14 Å for the V(CN), as found in this investigation, are compared to those of pure NbC(4.44 Å), NbN (4.38 Å), VC (4.17 Å) and VN (4.13 Å) [3], it becomes clear that the Nb(CN) precipitate probably contains very little, or practically no vanadium while the V(CN), on the other hand, cannot contain any significant amounts of niobium. It seems, therefore, that in this alloy an almost complete seperation of the niobium and vanadium into their respective carbon-rich and nitrogen-rich precipitates has occurred.

Furthermore, at 600°C, and to a smaller degree at 700°C, this particular alloy is understabilised and only at 800°C are the grain boundaries completely free from $M_{23}C_6$. No σ-phase formation could be found by the electron diffraction analysis but this does not exclude the possibility of σ-phase formation entirely as any σ-phase may have been overlooked due to the selective nature of electron diffraction analysis. From Bungardt and Lennartz [2], however, the amount of σ-phase in this alloy is expected to be relatively small and in some cases is only formed after prior cold work or during a heat treatment under stress.

Incoloy 800: mill-annealed

Results

The term "mill-annealed" is derived from the ASTM specifications for Incoloy 800 after a recrystallisation heat treatment at 980°C. The room temperature mechanical properties of the mill-annealed structure upon further long term annealing at 600 to 800°C, are shown in fig. 4 while in figs. 5 and 6 some of the corresponding microstructure are shown. At 600°C there is a decided strengthening effect upon annealing while at 700 and 800°C a decrease in strength properties has occurred after only a relatively short period of annealing.

Fig. 5 (a) shows the microstructure in the mill-annealed condition. The twin grain boundaries are entirely free from any precipitates while the high-angle grain boundaries contain only relatively few precipitates. These precipitates were identified as $M_{23}C_6$ which was probably not entirely dissolved by the mill-annealing heat treatment, which normally is sufficient to dissolve most of the $M_{23}C_6$. The $M_{23}C_6$ on the twin grain boundaries is less stable [4] and has dissolved entirely. The smaller and evenly spaced precipitates in the matrix could be identified as Ti(CN) with a lattice parameter of about 4.26 Å.

After further annealing for loo hours at 6oo°C, some marked changes in the microstructure are evident, as shown in fig. 5 (b). Extensive precipitation of $M_{23}C_6$ has occurred on both the high-angle grain boundaries and on the twin grain boundaries. In some cases the $M_{23}C_6$ on the high-angle grain boundaries appears as an almost continuous film while the $M_{23}C_6$ precipitates on the twin grain boundaries have nucleated as discrete seperate particles and these are much smaller in size as those on the high angle grain boundaries. Upon further annealing to lloo hours at 6oo°C (see fig. 5 (c)) the structure does not change very much. The $M_{23}C_6$ on the high-angle grain boundaries and on the twin grain boundaries has not coarsened noticeably and the appearance of the Ti(CN) remains unchanged.

After loo hours at 7oo°C the structure was very similar to that after lloo hours at 6oo°C ie. $M_{23}C_6$ precipitates present on both the high-angle grain boundaries as well as on the twin grain boundaries with the Ti(CN) dispersed throughout the matrix. Upon further annealing for 3oo hours at 7oo°C, however, it was found that practically all the $M_{23}C_6$ precipitates on the twin grain boundaries had disappeared and that the precipitates on the high-angle grain boundaries had started to coarsen, as shown in fig. 6 (a). This was especially noticeable after further annealing to 1100 hours at 700°C and also at 800°C (figs. 6 (b)-(d)) by the gradual disappearance of the seemingly continuous films of $M_{23}C_6$ on the high-angle grain boundaries and the formation of discrete ellipsoidal particles as shown in fig. 6 (d). The coarsening of $M_{23}C_6$, which is controlled by the diffusion of chromium through the matrix at these temperatures and occurs relatively rapidly above 700°C [5], would cause the breakdown of the grain boundary films because of a decrease in total surface energy and secondly, would also cause the transport of material from the less stable precipitates on the twin grain boundaries to the more stable precipitates on the high-angle grain boundaries, thus leading to the dissolution of the $M_{23}C_6$ on the twin grain boundaries.

Discussion

Incoloy 800 is principally a solid solution hardened alloy and consequently the carbides usually found in this material ($M_{23}C_6$ and TiCN) normally do not contribute significantly to the strengthening of this alloy. The presence of extensive precipitation of $M_{23}C_6$ on the grain boundaries can, however, lead to some changes in mechanical properties. The steady increase in strength during annealing at 600°C is probably related to the formation of local-ordered regions in the form of pre-precipitation zones of the γ'-(Ni_3(TiAl)) precipitate.

Such strengthening has been found on a number of occasions [4] in alloys that contain at least o.3 % Ti and o.4 % Al. Recently, a pairing of dislocations in such a strengthened alloy has been observed [6] which is a possible sign of local ordering or pre-precipitation zones and actual γ 'precipitates have been identified in alloys containing more than o.8 % Ti [6]. Therefore, it seems likely that the increase in strength observed during annealing at 6oo°C is related to this phenomenon of hardening by pre-precipitation zones. These will not be made visible by the technique of carbon extraction replicas until they have formed proper precipitates and have grown to a larger size.

No strengthening by this pre-precipitation phenomenon is expected at 7oo or 8oo°C as the upper solubility temperature limit for γ' in these alloys is only 62o°C for a titanium content of o.32 % Ti [4]. Less clear, however, is the drop in strength and the slight increase in ductility during annealing of this alloy at 7oo and 8oo°C. There is no doubt that this is somehow related to the extensive precipitation of $M_{23}C_6$ on the grain boundaries which can lead to some intergranular weakening but usually causes a decrease in ductility and not a slight increase as found here. It may, of course, be possible that at higher temperatures, where intergranular fracture processes predominate, such a decrease in ductility may be found after annealing at 7oo and 8oo°C. At 6oo°C the effects of the $M_{23}C_6$ precipitates are probably overshadowed by the strengthening caused by the pre-precipitation zones of γ'.

In general, the Ti(CN) precipitates do not play an active role in these alloys, altough some pinning of dislocations on their slip planes by these precipitates has been found to occur [6]. Comparison of the lattice parameter of about 4.26 Å for this precipitate, to that of pure TiC (4.32 Å) and TiN (4.24 Å) [3] would indicate a slightly nitrogen-rich carbo-nitride.

Incoloy 8oo: solution treated

Results

The tensile properties of the Incoloy 8oo, solution treated and then annealed at 7oo and 8oo°C, are shown in fig. 7 while in fig. 8 some of the corresponding microstructures are shown. In general, there was a very slight increase in ductility and yield strength and a slight decrease in tensile strength. The absolute strength levels are, however, well below those of the mill-annealed material and this is probably due to the much larger grain size of the solution treated material.

Fig. 8 (a) shows the microstructure of the material in the solution treated condition. Except for a very few isolated Ti(CN) particles, all the precipitates have been dissolved. After annealing for 1oo hours at 7oo°C, however, extensive precipitation of $M_{23}C_6$ has already occurred on the grain boundaries and a fairly fine precipitate has appeared in the matrix, as shown in fig. 8 (b). This latter precipitate was identified once again as a Ti(CN) precipitate but with a lattice parameter of about 4.3o Å, which is slightly larger than in the mill-annealed condition. Upon further annealing at 7oo and 8oo°C (figs. 8 (c) and (d)) the structure did not change markedly and some coarsening of both the $M_{23}C_6$ on the grain boundaries and the Ti(CN) is noticeable.

Discussion

The lattice parameter of the fine Ti(CN) indicates a slightly carbon-rich carbo-nitride which would mean that a redistribution of the carbon in the alloy had occurred after the solution treatment with more carbon in the matrix precipitate than was the case in the mill-annealed condition. This precipitate also has a different morphology than the nitrogen-rich carbo-nitride with small rods or needles being observed very frequently. Pinning of dislocations on their slip planes by these Ti(CN) precipitates would therefore be more likely than was the case in the mill-annealed material.

The smaller weakening effects of the $M_{23}C_6$ precipitate in the solution treated condition could be due to the much larger grain size resulting in less grain boundary area per unit volume than in the mill-annealed condition. Once again the slight increase in ductility after precipitation of the $M_{23}C_6$ cannot be explained satisfactorily unless the reason given above is correct.

Inconel 718

Results

The tensile properties of Inconel 718, solution treated and then aged at 6oo to 8oo°C, are shown in fig. 9 while in figs. lo and 11 some of the corresponding microstructures are shown. At 600°C marked strengthening has occurred, continuing even after 1100 hours at 600°C. At 700°C a similar strengthening has also taken place but after about 100 hours a gradual decrease in strength is found whereas at 800°C overageing has occurred after a relativ-ely short time and high strength levels are not attained. It is also clear that the ductility is impaired to a greater degree by ageing at 700°C than it is by a fully hardened structure, aged at 600°C, or by a fully overaged structure, aged at 800°C.

In fig. 10 (a) the structure of the solution treated material is shown. It is obvious that, altough the solution treatment temperature of 980°C was sufficient to fully soften the material, it was not high enough to dissolve all of the precipitates. The undissolved precipitates consisted mainly of the smaller evenly spaced particles, which were identified as M_6C, and a few large and angular particles as shown at the top of fig. 10 (a). These latter particles were identified by both electron diffraction and electron microprobe analysis to consist largely of a niobium-rich carbon-rich (NbTi)(CN) precipitate with a few titanium-rich nitrogen-rich (NbTi)(CN) particles interspersed at random, as shown by the electron back scatter micrograph in fig. 10 (b). The light coloured precipitates are the niobium-rich particles while the darker ones are the titanium-rich precipitates. (The circular array of diamond-shaped impressions are hardness indentations.) The niobium-rich particles were found to consist of about 70 % Nb and only about 4 % Ti. A small amount of molybdenum was also found in these precipitates but iron, chomium and nickel were totally absent.

These carbides, which have a size up to 6 microns in diameter and appear grey on an optically polished section, remove a considerable amount of niobium from the matrix. This precipitate is normally only taken into solution above lo5o^{o}C [7, 8]. The other massive precipitate, mainly TiN, is kown to be very stable and to remain out of solution practically up to the melting point of the alloy [7]. This precipitate was found to contain very little niobium and no molybdenum, iron, chromium or nickel and appears pink on a optically polished section.

Upon ageing at 6oo^{o}C the microstructure at first seemed to have remained unchanged although a marked degree of hardening had occurred, as shown by the extraction replicas in figs. lo (c) and (d). Examination of a thin foil specimen aged for lloo hours at 6oo^{o}C, however, produced evidence of an extremely fine precipitate in the matrix as shown in fig. 11 (a). The size of this precipitate is, therefore, below the limit of resolution of carbon extraction replicas. Fig. 11 (b) shows a diffraction pattern from the area shown in fig. 11 (a). This pattern is in the [211] orientation and shows superlattice reflections from an ordered DO_{22} body centered tetragonal phase. This phase in Inconel 718, is a disc-shaped metastable bct phase with composition Ni_3 (NbAlTi), and is often called γ' or γ'' although the structure differs from the normal fcc-γ' found in many other nickel-base alloys [8]. This coherent bct-γ' is known to cause the strengthening in Inconel 718 [9]. Ageing for loo hours at 7oo^{o}C produced the first visible microstructural changes in the extraction replicas, as shown in fig. 12 (a). (Note the higher magnification).

The grain boundaries are still relatively free from any precipitates but the grain interior is covered extensively by the bct-γ' precipitates, which have grown considerably if compared to those at 600°C. In addition to this phase, some M_6C particles are also present in the structure. Upon further ageing for 300 hours at 700°C, the bct-γ' phase has coarsened considerably and a film of NbC is found on the grain boundaries, as shown in fig. 12 (b). Such a film of NbC can impair the ductility and explains the rather low elongation values found in this alloy after ageing at 700°C, as shown in fig. 9. This is mainly the reason for the preference in practice for low solution treatment temperatures as higher temperatures would dissolve more of the massive niobium-rich NbTi(CN) phase, making it available for later nucleation on the grain boundaries as a film. Upon further ageing to 1100 hours at 700°C, the bct-γ' phase has coarsened still further and the first signs of the stable orthorhombic Ni_3Nb phase have appeared in the form of large discs, as shown in fig. 12 (c). (Note the difference in magnification between figs. 12 (a), (b) and (c)). This stable orthorhombic phase forms partly from the metastable bct-γ' phase upon overageing. This is particularly clear in fig. 12 (c) where the bct-γ' phase has disappeared at those locations where the first Ni_3Nb discs have nucleated. The NbC films on the grain boundaries, however, also play on active role in the transformation as shown by the preference for nucleation of the Ni_3Nb on these grain boundary films and their later dissolution during further growth of the stable phase.

Ageing at 8oo°C produces a virtually overaged structure after only 1oo hours, as shown in fig. 12 (d). In between the large discs of Ni_3Nb, some last traces of the finer bct-γ' phase are still visible. Upon further ageing at 8oo°C, however, these disappear quickly and the strength drops to almost that of the solution treated structure. After 11oo hours at 8oo°C, therefore, the structure consists of relatively coarse plates of Ni_3Nb with a few M_6C particles still present in between, as shown in fig. 12(e). (The Ni_3Nb plates had reached such a size that they could not be extracted by the carbon films any more and the replica is therefore similar to a surface replica.) Fig. 12 (f) shows an interesting feature of the Ni_3Nb plates. Apparently they are able to interpenetrate each other, causing the meeting point to be pinched off. The behaviour of the NbC on the grain boundaries is also of interest. After 1oo hours at 8oo°C the grain boundary films of NbC have broken up to form a chain of ellipsoidal particles while after 11oo hours even these have disappeared, leaving the grain boundaries free from any precipitates. This causes a higher ductility than that at 7oo°C where grain boundary films are present.

Discussion

In general, the change in tensile properties during ageing could be correlated quite well with the observed microstructural changes. Strengthening of this alloy is produced by the finely divided bct-γ' phase. Paulonis et al [9] have found that strengthening is mainly due to the coherency strains around these particles and that they can become sheared during deformation. Loss of strength of this alloy upon overageing at 7oo and 8oo°C could be due to two factors viz. the bct-γ' precipitate coarsens and loses its effectiveness as a strengthening phase or the volume fraction of the bct-γ' phase decreases as transformation to Ni_3Nb occurs. The decrease in strength properties during overageing at 700°C would seem to indicate that both of these factors contribute in turn. Between 100 and 700 hours softening occurs mainly due to coarsening of the bct-γ' precipitate and upon further ageing an accelerated softening is found due to the formation of the Ni_3Nb precipitate.

The transformation from the bct-γ' phase to the stable orthorhombic Ni_3Nb has received little attention in the past. From figs. 12 (c)-(f) some informative observations could be made regarding this transformation. In the first place, the nucleation of the Ni_3Nb occurs preferentially at the NbC films on the grain boundaries and in the process these NbC films are broken up into seperate particles which soon change into ellipsoidal particles. As the Ni_3Nb plates grow into the grain interior the bct-γ' precipitates dissolve to provide the necessary material at the growth front. The NbC film on the grain boundary is, however, an equally important source of material for the further growth of the Ni_3Nb as fig. 12 (d) indicates. Here the grain boundary film has already been broken up and has started to dissolve, thereby supplying niobium to the Ni_3Nb, even before all the bct-γ' in the grain interior has dissolved. Finally, after further overageing, both the NbC and the bct-γ' have dissolved completely (fig. 12 (e)) to supply the rapidly growing Ni_3Nb with the necessary niobium. The breaking up of the NbC films on the grain boundaries, by the nucleating Ni_3Nb, has decidedly a favourable effect on the ductility as shown by the increase, once again, in elongation after 1100 hours at 700°C.

Instead of the hitherto stated transformation, bct-γ' ⟶ Ni_3Nb, it is therefore thought to be more correct to write the reaction as follows:

$$\left.\begin{array}{l}\text{bct-}\gamma' \\ \text{NbC (grain boundary film)}\end{array}\right. \longrightarrow Ni_3Nb$$

The exact role of each outgoing phase in the transformation is not entirely clear at this stage. However, after 1100 hours at 700°C (fig. 12 (c)) some grain boundary zones free from bct-γ' precipitates are found for the first time, even at those grain boundaries on which no Ni_3Nb plates have nucleated yet. This could possibly indicate that the transformation is preceeded by the dissolution of the bct-γ' phase near the NbC films on the grain boundary, which is then renucleated on the NbC in the form of Ni_3Nb. The NbC does not, however, act as a simple nucleation catalyst as the breaking up of the films and later dissolution of the NbC shows that it must participate in the reaction. The carbon that is released upon the dissolution of the NbC films probably reprecipitates in the form of another carbide such as M_6C or MC.

Conclusions

Steel 4988

1. At 600°C some hardening of the alloy occurs by the precipitation of a very fine nitrogen-rich V(CN) phase.

2. At 700 and 800°C these V(CN) precipitates coarsen rapidly, losing their effectiveness as a strengthening phase.

3. The relatively large carbon-rich Nb(CN) precipitates do not seem to play a role in determining the response to ageing of this alloy.

4. At 600 and 700°C the alloy is understabilised and only at 800°C are the grain boundaries free from any $M_{23}C_6$.

Incoloy 800 (mill-annealed and solution treated)

1. This alloy is strengthened considerably during ageing at 600°C, probably due to the formation of pre-precipitation zones of Ni_3 (TiAl).

2. The Ti(CN) precipitate in this alloy does not seem to cause any hardening.

3. At 600°C $M_{23}C_6$ is found on both twin grain boundaries and high angle grain boundaries. At higher temperatures the $M_{23}C_6$ precipitate on high angle grain boundaries coarsens at the expense of that on the twin grain boundaries.

4. The Ti(CN) precipitate in the solution treated and aged structure has a different morphology and a slightly larger lattice parameter than in the mill-annealed and aged structure.

Inconel 718

1. Ageing of this alloy at 6oo°C for 11oo hours more than doubles the yield strength from that of the solution treated condition.
2. At 7oo°C a similar hardening response to ageing is found but after 1oo hours overageing causes a drop in strength. At 8oo°C overageing is very rapid and high strength levels are not attained.
3. Strengthening of this alloy occurs by the precipitation of a metastable coherent bct-γ' phase and upon overageing the stable orthorhombic Ni_3Nb phase forms.
4. The NbC phase forms as a film on the grain boundaries at 7oo°C, causing a pronounced loss in ductility.
5. Nucleation of the Ni_3Nb phase occurs preferentially on the NbC films on the grain boundaries and both the NbC and the bct-γ' phases dissolve in the course of the transformation to Ni_3Nb.
6. The breaking up of the NbC films on the grain boundaries into ellipsoidal particles during the nucleation of the Ni_3Nb phase, causes an increase in ductility.

Acknowledgements

I would like to thank Dr. W. Hein for the microprobe analysis of the Inconel 718 alloy and Dr. K. Ehrlich for arranging the thin foil electron microscopy of the same alloy. I would further like to extend my appreciation to the South African Atomic Energy Board for making my stay at the Karlsruhe Nuclear Research Center financially possible.

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Table I Composition of Alloys (wt %)

Alloy Melt no.	C	Si	Mn	P	S	Cr	Ni	Co	Mo	V	Cu	Nb	N	Ti	Al	Ta	B
4988 M166/66	0.08	0.29	1.10	0.19	0.10	16.1	13.6	0.05	1.30	0.75	0.05	0.87	0.10				1-2 ppm
Incoloy 800 M209/68	0.03	0.46	0.67	0.006	0.003	20.5	31.6	0.10	0.05		0.10		0.06	0.32	0.40		2-3 ppm
Inconel 718 M167/66	0.04	0.30	0.19	0.005	0.005	18.8	52.5	0.05	3.0		0.04	5.0		0.90	0.50	0.13	6 ppm

Table II Initial Heat Treatments and Resulting Grain Sizes

Alloy	Heat Treatment	Grain Size µm
4988-aged	1050°C for 1 hr-WQ, aged 750°C for 3 hr-AC	8
Incoloy 800 mill-annealed	980°C for 1 hr-WQ	14
Incoloy 800 solution treated	1150°C for 1 hr-WQ	42
Inconel 718 solution treated	980°C for 1 hr-AC	11

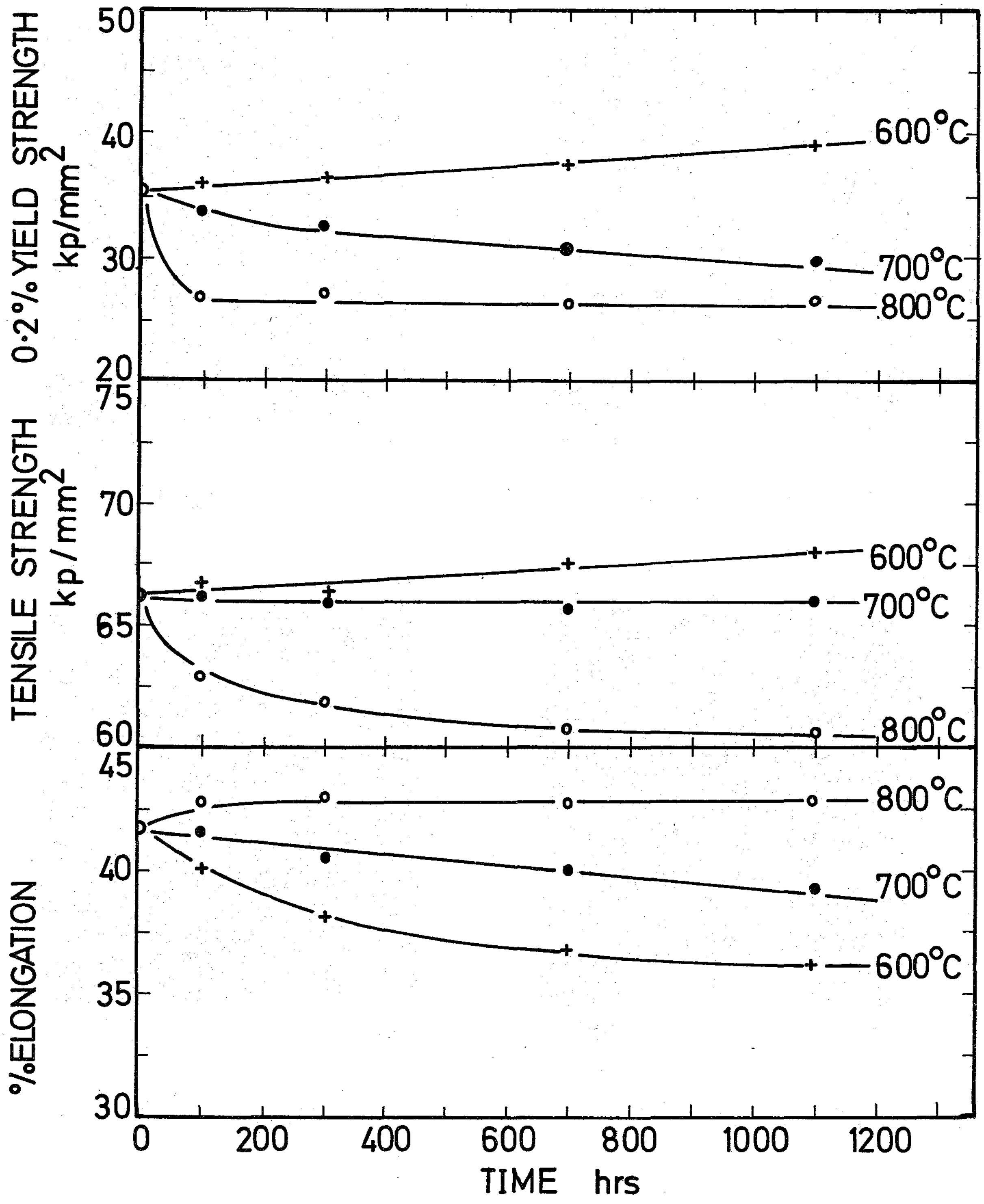
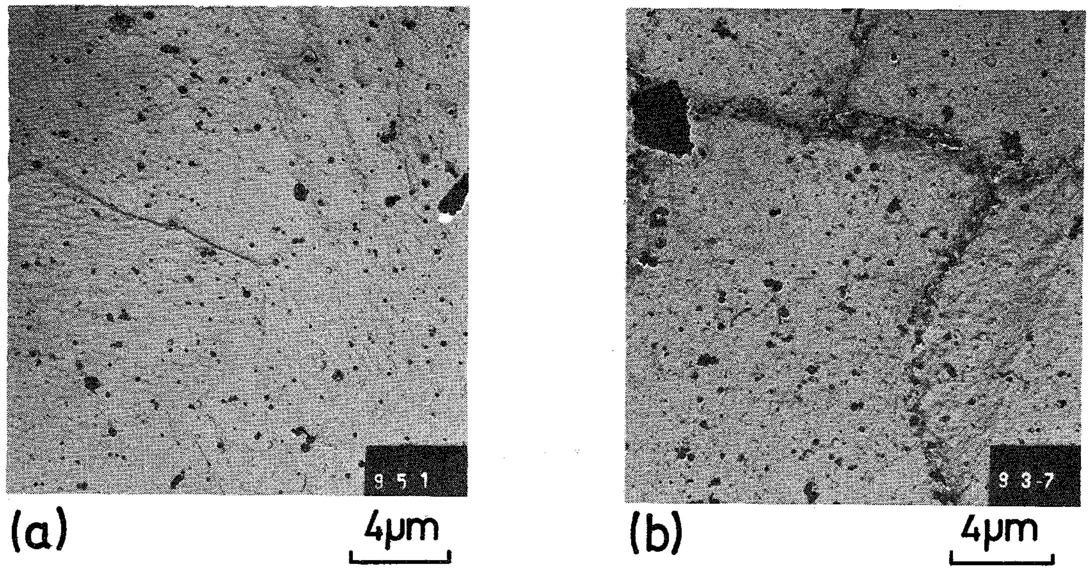


Fig. 1

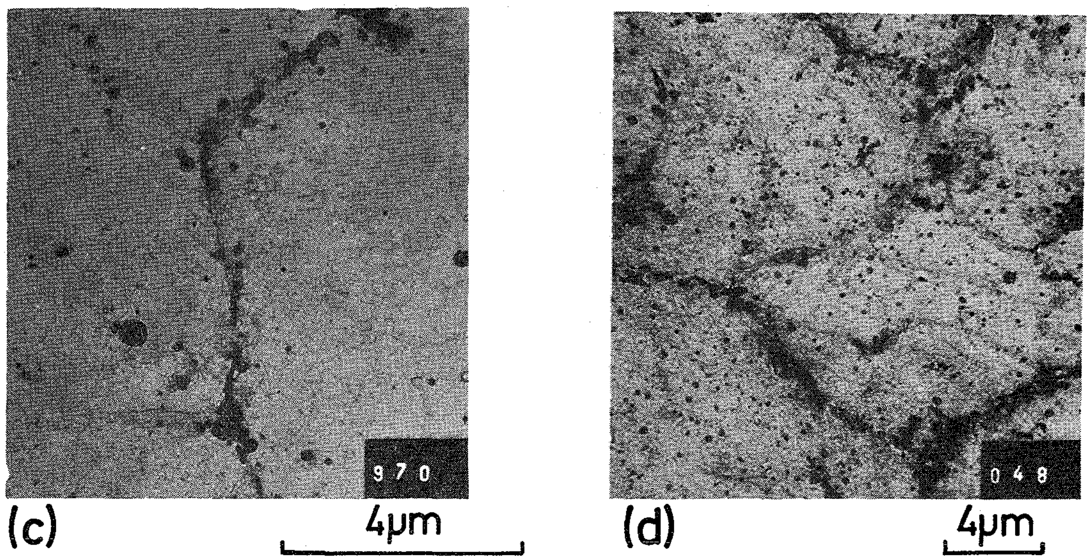
STEEL 4988 solution treated and aged for 3 hrs at 750°C. Room temperature mechanical properties after further ageing.

STUMPF 1970



(a) no further ageing

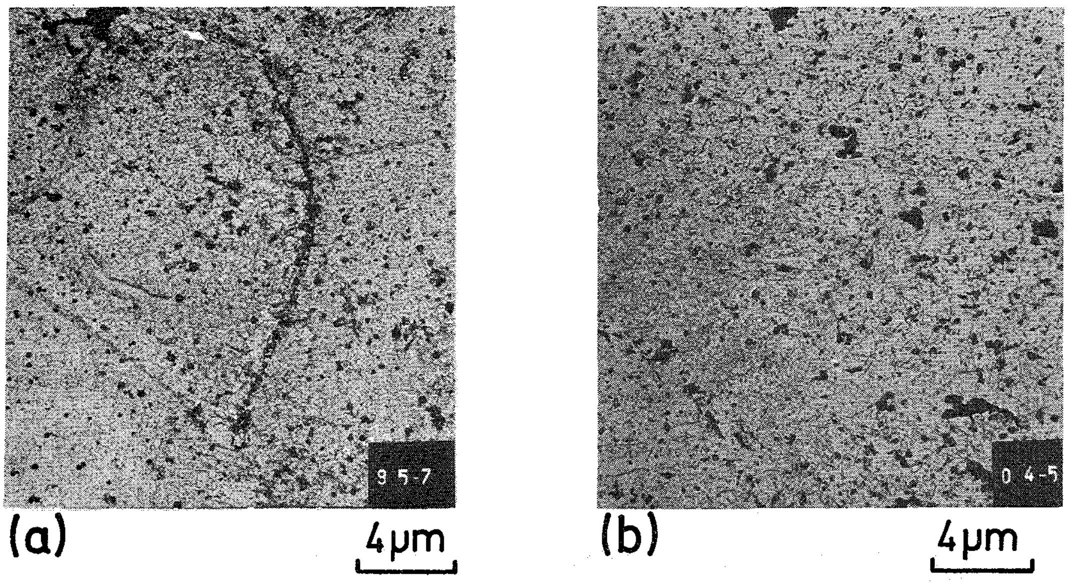
(b) 600°C - 300 hrs



(c) 600 °C - 300 hrs

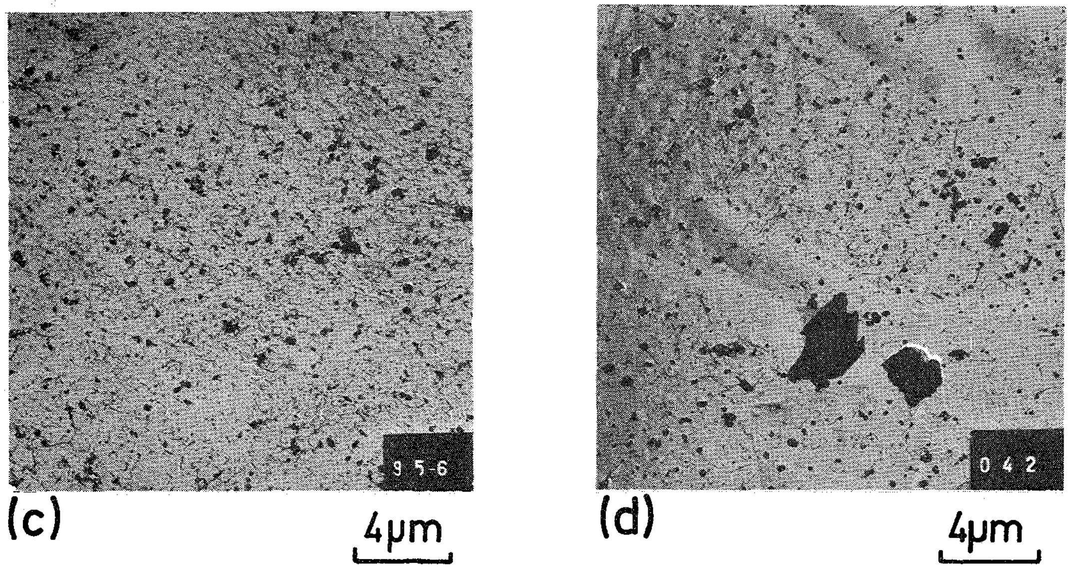
(d) 600°C - 1100 hrs

Fig. 2	STEEL 4988 solution treated and aged for 3 hrs at 750 °C before ageing further as shown	STUMPF 1970



700°C - 100 hrs

700°C - 1100 hrs



800°C - 100 hrs

800°C - 1100 hrs

Fig. 3

STEEL 4988 solution treated and aged for 3 hrs at 750°C before ageing further as shown

STUMPF
1970

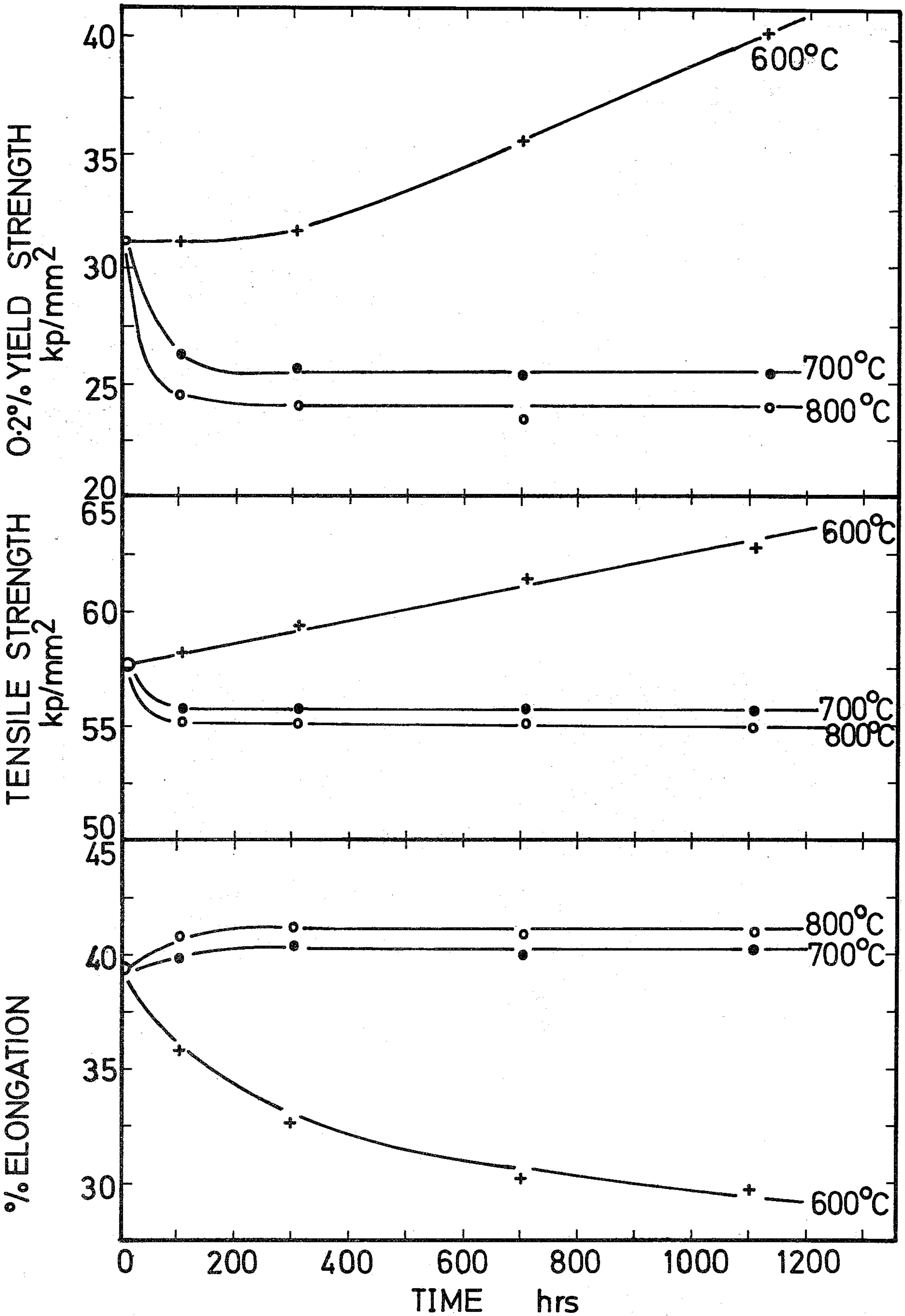
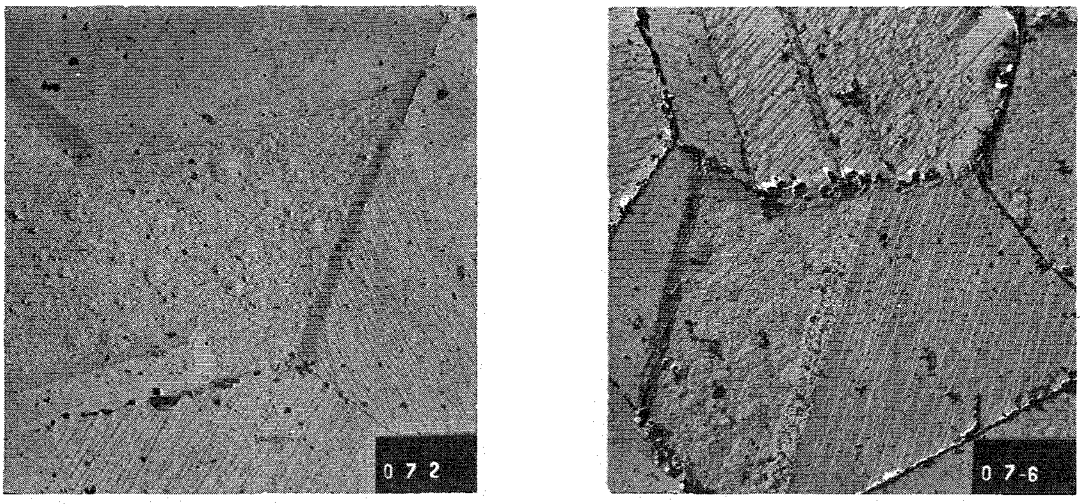


Fig. 4	INCOLOY 800 mill-annealed. Room temperature mechanical properties after further ageing as shown	STUMPF 1970

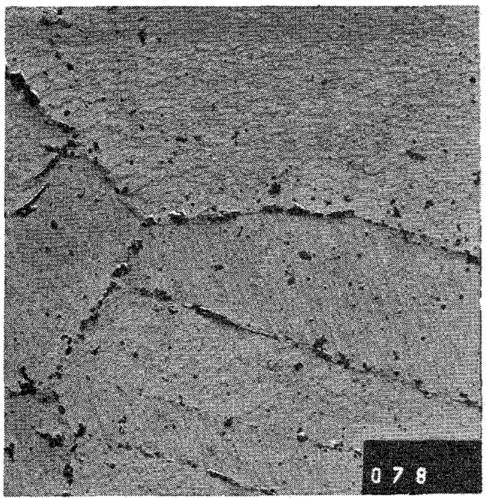


(a) 4μm

mill-annealed

(b) 4μm

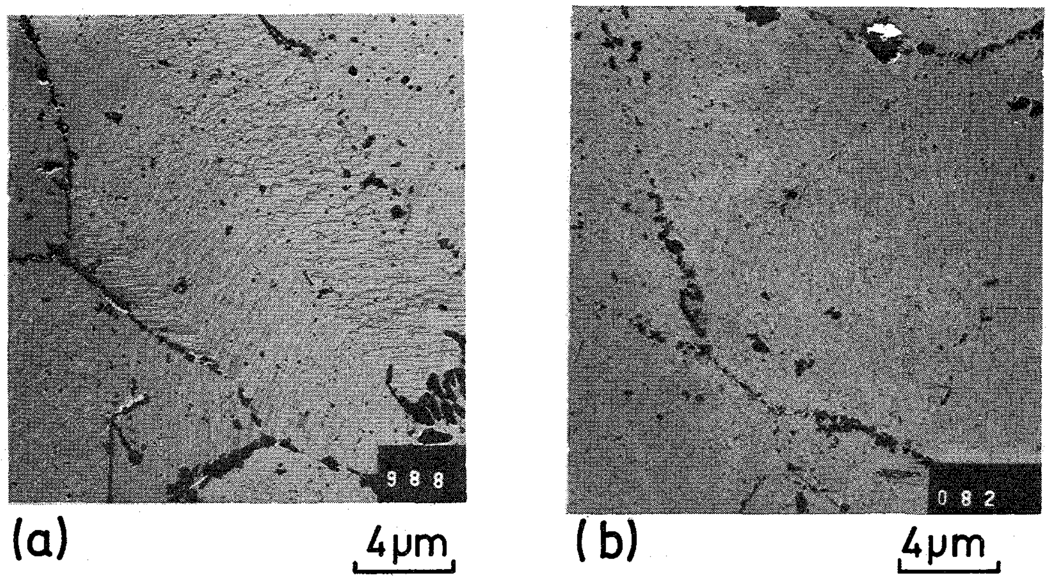
600°C - 100 hrs



(c) 4μm

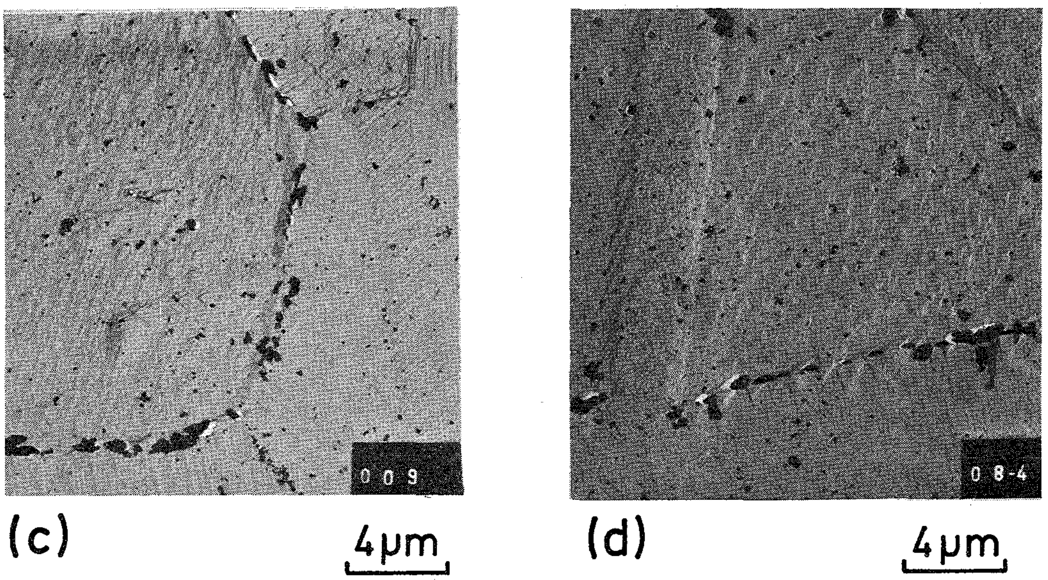
600°C - 1100 hrs

Fig. 5	INCOLOY 800 mill-annealed and aged further as shown	STUMPF 1970



700°C - 300 hrs

700°C - 1100 hrs



800°C - 300 hrs

800°C - 1100 hrs

Fig. 6

INCOLOY 800 mill-annealed and aged further as shown

STUMPF 1970

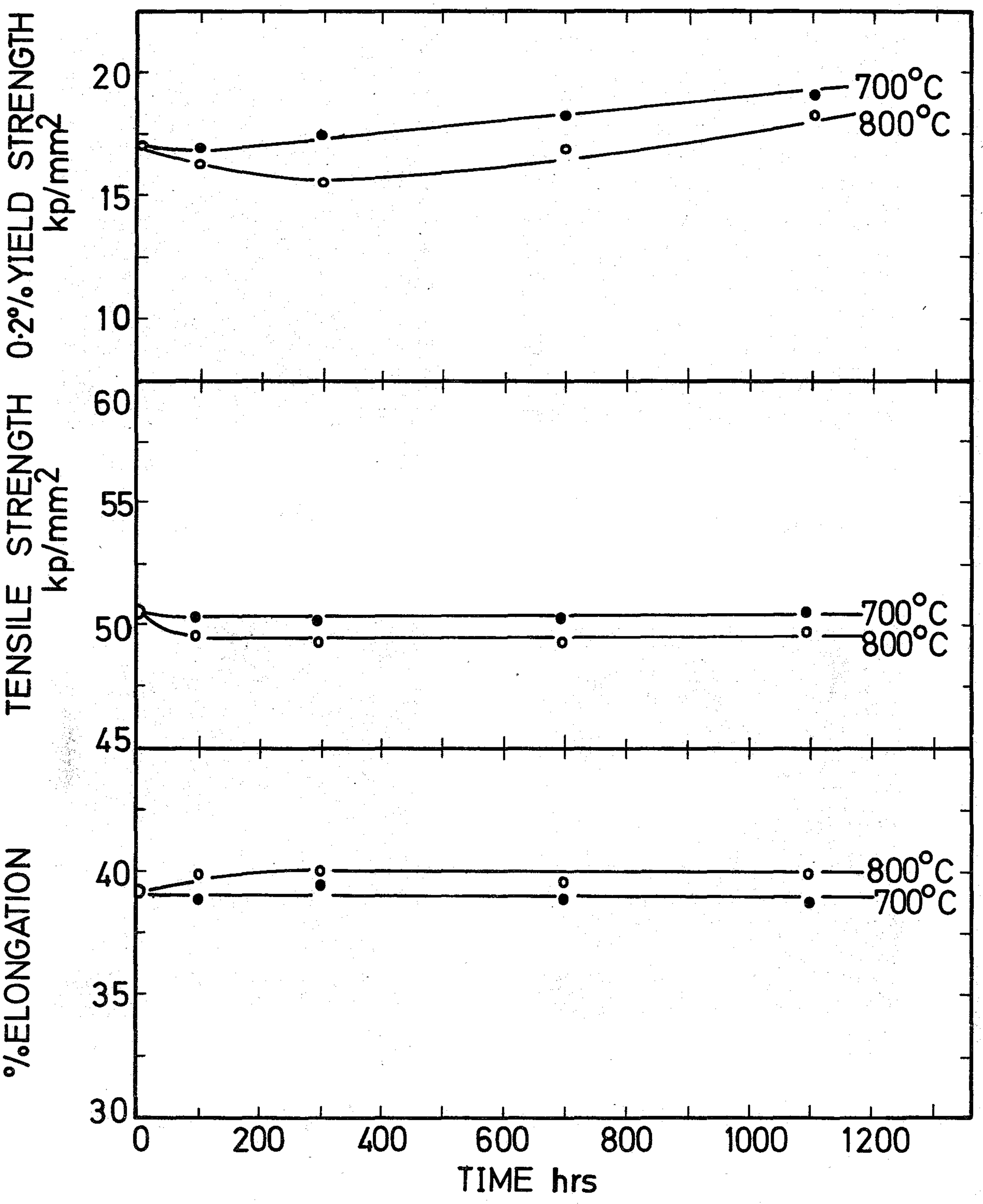
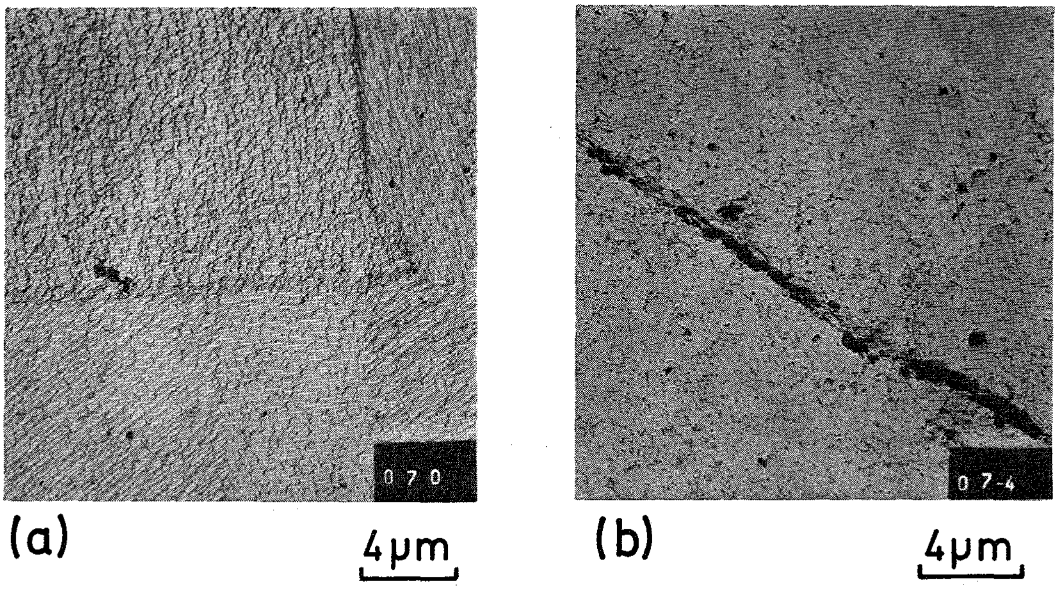
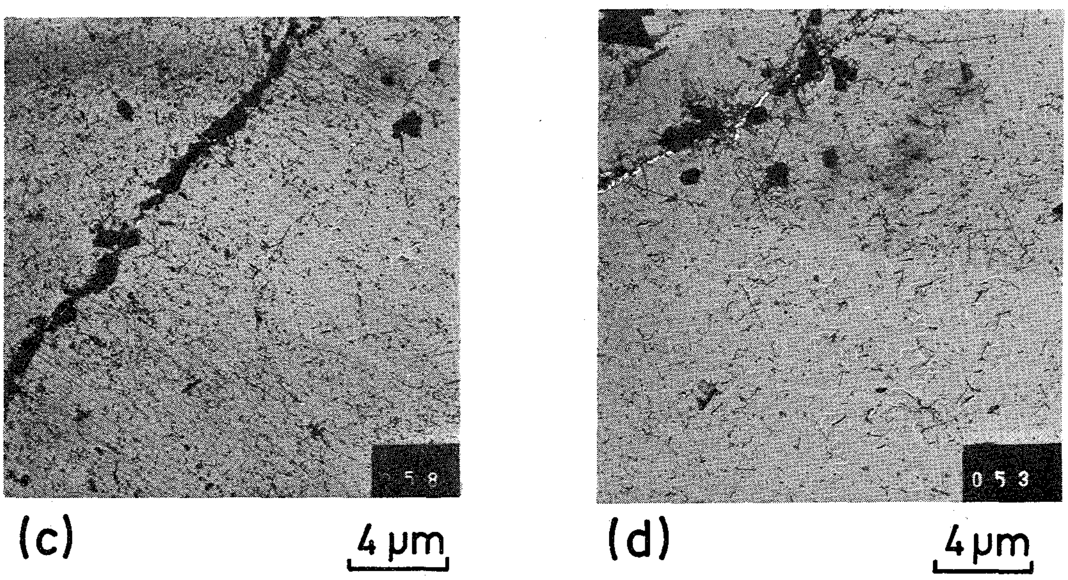


Fig. 7	INCOLOY 800 solution treated. Room temperature mechanical properties after further ageing as shown	STUMPF 1970



(a) solution treated

(b) 700° C - 100 hrs



(c) 700° C - 1100 hrs

(d) 800° C - 1100 hrs

Fig. 8

INCOLOY 800 solution treated and aged further as shown

STUMPF 1970

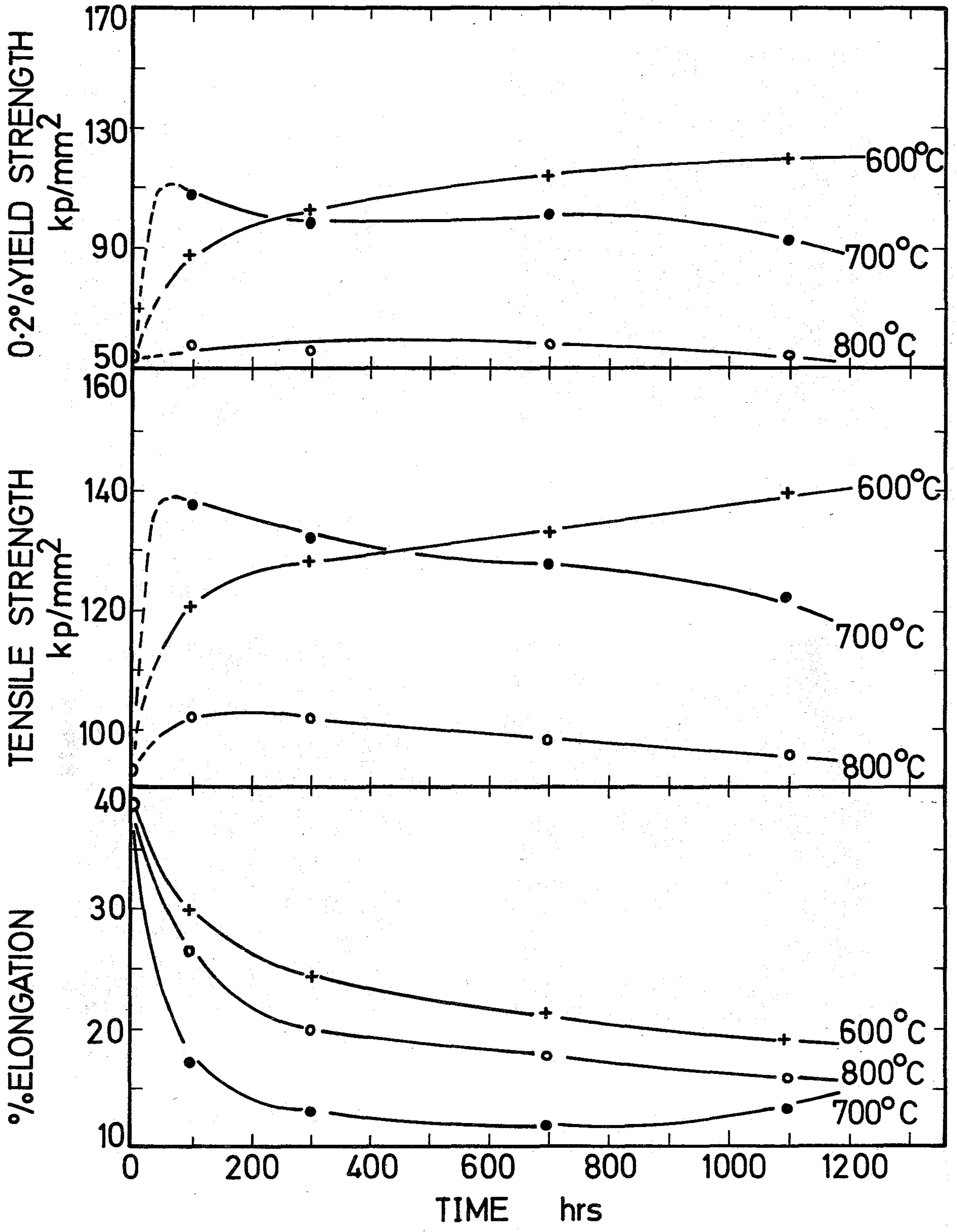
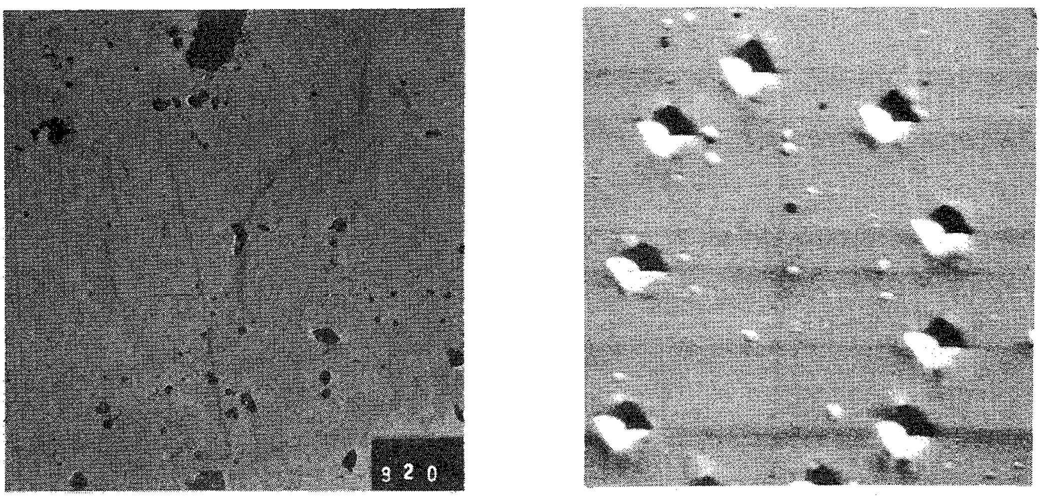


Fig. 9

INCONEL 718 solution treated.
Room temperature mechanical properties
after further ageing as shown.

STUMPF
1970

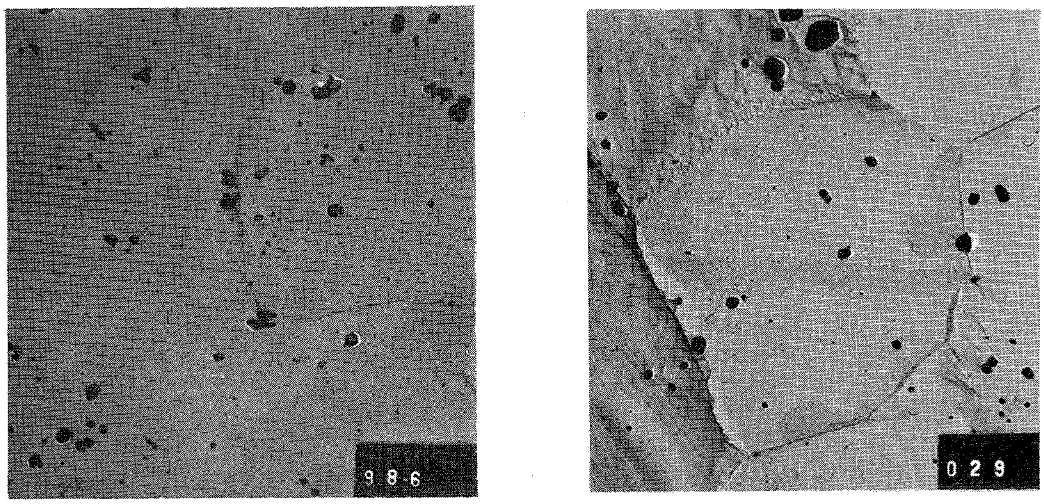


(a) 4 μm

solution treated

(b) 40 μm

solution treated
(electron back scatter
micrograph)



(c) 4μm

600 °C - 100 hrs

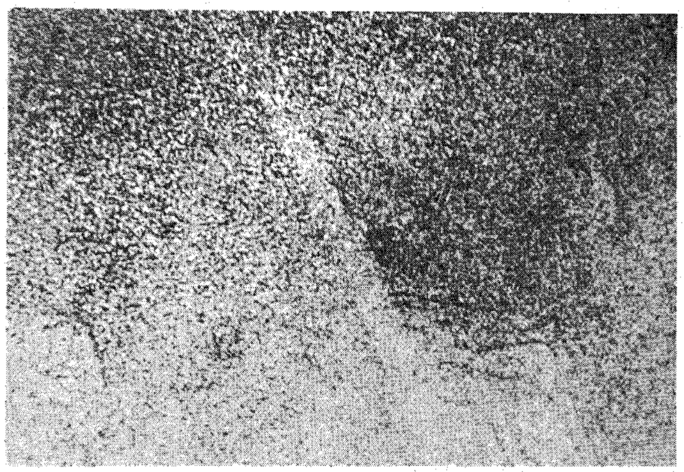
(d) 4μm

600 °C - 1100 hrs

Fig. 10

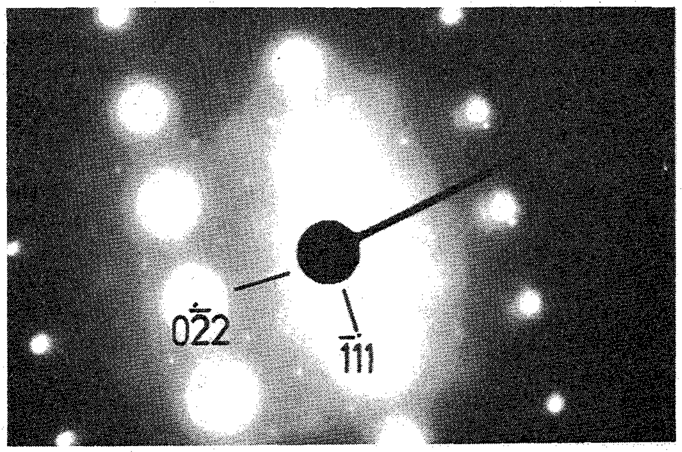
INCONEL 718 solution treated
and aged further as shown

STUMPF
1970



(a)

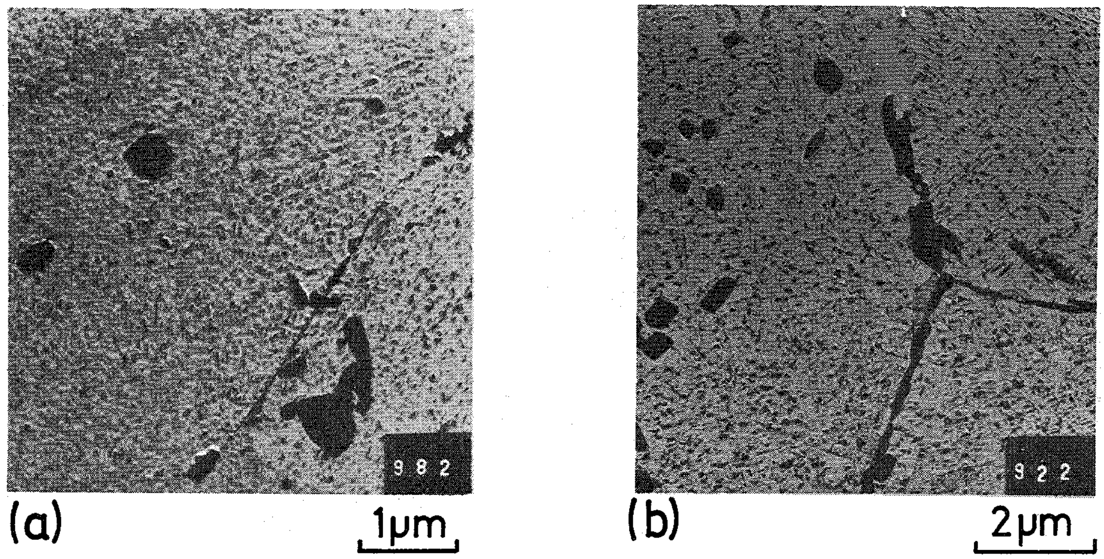
Thin foil micrograph



(b)

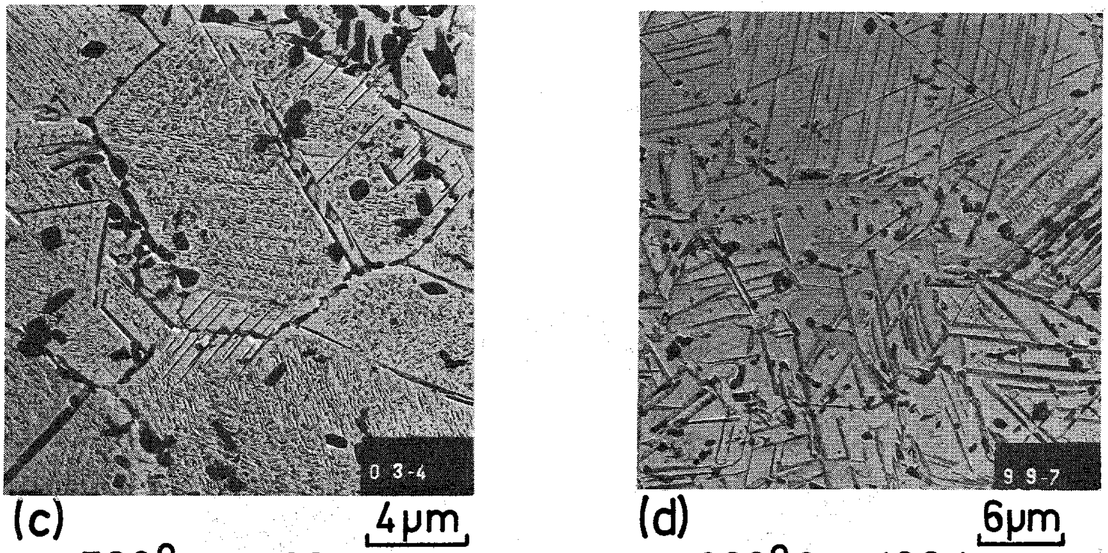
Electron diffraction pattern.
[211] orientation

Fig. 11	INCONEL 718 solution treated and aged 1100 hrs at 600 °C	STUMPF 1970



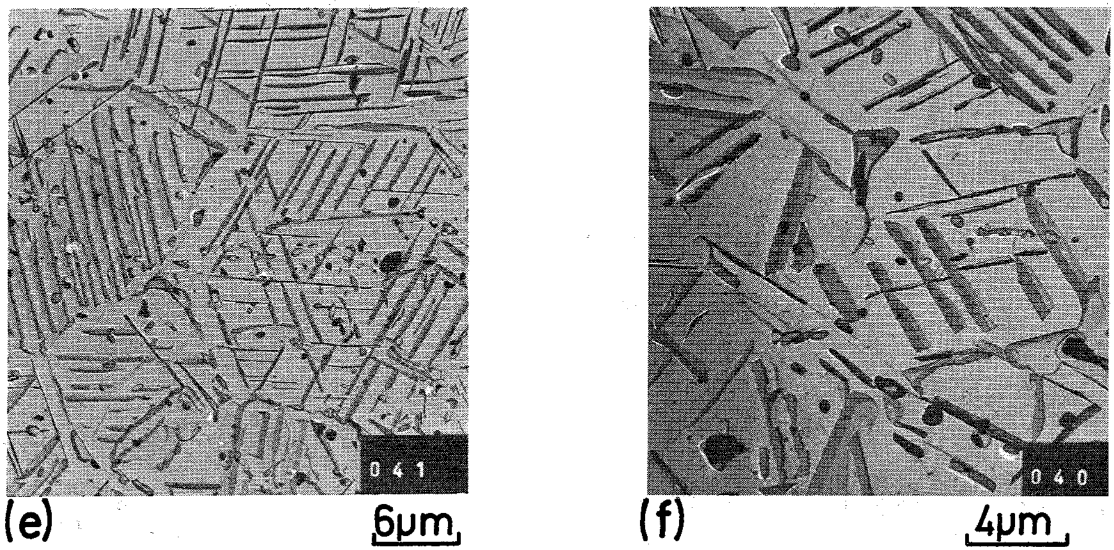
(a) 700° C - 100 hrs

(b) 700°C - 300 hrs



(c) 700° C - 1100 hrs

(d) 800° C - 100 hrs



(e) 800° C - 1100 hrs

(f) 800° C - 1100 hrs

Fig. 12	INCONEL 718 solution treated and aged further as shown	STUMPF 1970