THE EFFECT OF $\sigma$ PHASE ON THE MECHANICAL PROPERTIES IN Ni-Cr-Co BASE WROUGHT SUPERALLOYS

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ABSTRACT

The effect of $\sigma$ phase on the alloy properties is closely related to its quantities in alloys. $\sigma$ phase precipitated in trace and small quantities after long time exposure at 850°C had no significant effect on the mechanical properties of the alloys whereas $\sigma$ phase in large quantities lowered the tensile properties at room temperature, impact toughness, stress-rupture and creep properties and fracture toughness markedly. Tensile properties at elevated temperature and high frequency fatigue property, however, seemed to be slightly affected. It appears that the hard $\sigma$ phase impeded the movement of dislocations and sliding at room temperature and led to brittle fracture, but $\sigma$ phase possessed some plasticity at elevated temperature. The transition temperature, above which the brittle influence of $\sigma$ on the experimental alloys disappeared, may be 800-850°C.

INTRODUCTION

There is a common tendency for some complex superalloys to precipitate TCP phases, such as $\sigma$, $\mu$ and Laves etc., during long time exposure. $\sigma$ phase belongs to the tetragonal crystal system, is hard and brittle, and exists mostly in the form of
flakes. In most references $\sigma$ phase is indicated to be harmful to tensile and stress-rupture properties, sometimes even catastrophic (1, 2, 3, 4). But other show that $\sigma$ phase is not detrimental to stress-rupture properties in alloys such as Inco 713C (4,5).

In this work the relationship between the varying quantities of $\sigma$ phase in the alloy and the mechanical properties is systematically investigated, which appears not to have been done before. The mechanical properties studied include tension, stress-rupture, creep, fatigue and fracture toughness. A study of the influence of $\sigma$ phase on the deformation behaviour has also been carried out.

MATERIALS USED AND EXPERIMENTAL TECHNIQUES

The alloy system studied was Ni-Cr-Co base wrought superalloy, the composition of which was Ni-15Cr-15Co-5Al-4Ti-3.7Mo. The materials were melted in a vacuum induction furnace and remelted in a vacuum arc furnace, then deformed into bars. The heat treatment cycle consisted of 1190°C, 1.5 hrs, AC and 1100°C, 6 hrs, AC. $\sigma$ Phase precipitated during exposure at 750°C-950°C in the form of needle or Widmanstatten structure, and with fastest speed at 850°C-900°C.

The amounts of $\sigma$ phase in the alloys were designated in five grades, i.e. trace, small, medium, large and extra large quantity. Their metallographic characteristics are shown in Fig. 1 and their $\sigma$ phase contents are about $<0.05$ wt% for trace, 0.1-0.2 wt% for small, 0.3-0.5 wt% for medium, 0.6-1.0 wt% for large and > 1.2 wt% for extra large quantity. These were obtained from the heats with slightly modified compositions and different average $N_v$ - numbers after the same exposure at 850°C. In order to determine the effect of $\sigma$ phase only on mechanical properties, changes of properties caused by long time exposure have been taken into consideration. A comparative method was therefore adopted. With a few heats, in which $\sigma$ phase did not precipitate, the normal decline of properties after exposure was determined and then it was compared with those, in which $\sigma$ precipitated in different amounts.
The study of the effect on deformation behaviour was conducted with special tensile specimens, the surfaces of which were polished and eroded, in a vacuum tensile test machine. After some deformation, the interaction between slip lines and $\sigma$ phase was observed. Testing of mechanical properties and metallographic study with optical and electron microscope were done using ordinary techniques.

**EXPERIMENTAL RESULTS**

Fig. 2 shows the change of tensile properties at room temperature after exposure at 850°C. It can be seen that the ultimate tensile strength, elongation and reduction of area of the heats without $\sigma$ phase after exposure decreased. This could be considered to be due to the normal structural change of the alloy during exposure, i.e. precipitation of $\text{M}_{23}\text{C}_6$ carbide at the grain boundary, degradation of $\text{M}_{3}\text{C}$ carbide and coarsening of $\gamma'$ phase etc. In specimens containing trace and small quantities of $\sigma$, the $\sigma$ phase had no significant effect on tensile property, but the presence of large quantity of $\sigma$ decreased it considerably. Compared with the values before exposure, the decrease in ultimate tensile strength and elongation would be 22% and 99% respectively. The effect of $\sigma$ phase on impact toughness at room temperature is also shown in Fig. 2. In a similar way, the effect of $\sigma$ in trace or small amounts was not significant, but a marked decrease of impact occurred with a great amount of $\sigma$ phase. An examination of the fractured specimens showed
Fig. 2 Effect of σ on the tensile properties and impact toughness at room temperature.

That whereas specimens without σ or with small quantity of σ after exposure cracked along grain boundaries, specimens with large quantities of σ showed brittle fracture which was transgranular and along the interface between σ phase and the matrix (Fig. 3).

The effect of σ phase on tensile properties at elevated temperature is shown in Table 1. Elongation and reduction of area raised slightly after exposure, while ultimate tensile strength decreased a little. Large amounts of σ phase had no influence on tensile properties at elevated temperature, which was different from the case at room temperature. In the specimens tested at elevated temperature, the σ phase became bent (Fig. 3). This means that σ phase has some degree of plasticity at high temperature. The fracture may occur along grain boundaries or along σ phase.
Table I. Effect of $\sigma$ phase on $K_c$ at room temperature, tensile properties at 850°C and high frequency fatigue at 700°C

<table>
<thead>
<tr>
<th>Heat Exposure</th>
<th>Amount of $\sigma$ at r.t. strength</th>
<th>700°C fatigue for 10 cyc. $N/mm^2$</th>
<th>850°C tensile $N/mm^2$</th>
<th>%</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>none</td>
<td>none</td>
<td>--</td>
<td>--</td>
<td></td>
<td></td>
</tr>
<tr>
<td>850°C, 100h</td>
<td>none</td>
<td>73, 73</td>
<td>--</td>
<td></td>
<td></td>
</tr>
<tr>
<td>850°C, 150h</td>
<td>small</td>
<td>64, 70</td>
<td>--</td>
<td></td>
<td></td>
</tr>
<tr>
<td>none</td>
<td>none</td>
<td>--</td>
<td>431</td>
<td>80.5</td>
<td>11.6</td>
</tr>
<tr>
<td>850°C, 150h</td>
<td>none</td>
<td>--</td>
<td>382</td>
<td>--</td>
<td>1</td>
</tr>
<tr>
<td>850°C, 200h</td>
<td>none</td>
<td>--</td>
<td>362</td>
<td>76.9</td>
<td>24.9</td>
</tr>
<tr>
<td>none</td>
<td>none</td>
<td>130, 144</td>
<td>431</td>
<td>85.0</td>
<td>17.6</td>
</tr>
<tr>
<td>850°C, 150h</td>
<td>medium</td>
<td>62, 57</td>
<td>382</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>850°C, 200h</td>
<td>large</td>
<td>57, 47</td>
<td>--</td>
<td>81.2</td>
<td>18.4</td>
</tr>
<tr>
<td>850°C, 150h</td>
<td>medium</td>
<td>--</td>
<td>382</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>850°C, 150h</td>
<td>large</td>
<td>372-382</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

Compared with unexposed alloy specimens, the stress-rupture life and elongation of those without $\sigma$ or with trace and small quantities of $\sigma$ phase after exposure did not change considerably at 980°C. $\sigma$ Phase in large quantities decreased the rupture life to a great extent (about 60%) and increased elongation (Fig. 4). At 850°C, trace and small amounts of $\sigma$ had a slight effect on the stress-rupture life, whereas medium and large amounts after exposure at 850°C decreased the endurance life to even lower values (50 and 20 hrs. respectively). Their rupture life before exposure was originally very low (Fig. 4). Experiments proved that $\sigma$ phase dissolved gradually during the stress-rupture test at 980°C, but the process at 850°C was in the opposite direction, i.e. $\sigma$ precipitated continuously and made the alloy worse than before. The elongation of specimens with large amounts of $\sigma$ increased. The 850°C creep curve showed that the presence of $\sigma$ phase caused an increase of creep strains. An enhancement of creep rate at the secondary stage and an earlier oc-
Fig. 4. Effect of $\sigma$ on stress-rupture properties at 850°C and 980°C

currence of the third stage were caused by great quantities of $\sigma$ phase (Fig. 5). That was coincident with an increase of elongation and a reduction of life in the stress-rupture test. The fracture was similar to tensile fracture. In the stress-rupture specimens tested at 850°C, $\sigma$ phase was observed obviously to be zigzag or bent near the location of rupture (Fig. 8a).

The fracture toughness $K_t$ at room temperature of the alloys before exposure was 135-160 MNm$^{-3/2}$ as shown in table 1. After exposure the $K_t$ values of the specimens without $\sigma$ phase decreased to 60-75 MNm$^{-3/2}$. This is related closely to carbide precipitation at grain boundaries. Trace or small quantity of $\sigma$ phase had no further influence on $K_t$, but a large quantity caused it to decrease to
45-55 MNm⁻¹/². The fracture characteristics were similar to those of tensile specimens at room temperature.

Data from rotating bending fatigue tests at 700°C indicated that σ phase in medium quantity or less had no influence on the h.f. fatigue property (Table 1).

DISCUSSION

The experiments have demonstrated that σ phase in great quantities had a marked influence on the mechanical properties of these alloys at room temperature, especially ductility and impact toughness. Even if the strain was only 1.5-3%, brittle failure occurred along the interface between σ and the matrix. Electron microscopic observation on thin foils of used tensile specimens showed piling of dislocations and the presence of dislocation networks near the σ phase (Fig. 6). The very high dislocation density in the neighbourhood of σ phase caused severe stress concentration, which led to cracking. It can be seen that the dislocation movement was impeded by the hard and brittle σ phase, thus influencing the strain behaviour of the material. Deformation tests at room temperature under pressure showed that in the another case σ plates could sometimes also fracture under stress (Fig. 7).

Fig. 6. Dislocations in the neighbourhood of σ on the tensile specimen (δ = 3%) at r.t. (thin foil)

Fig. 7. Fracture of σ-plate in the sliding deformation tests (replica)
The alloys with large quantities of $\sigma$ phase had rather high ductility at elevated temperature ($850^\circ$C), contrary to room temperature, and a lot of slip lines on the surfaces of deformation specimens were observed. The $\sigma$ phase possessed some plasticity and became bent or zigzag by interaction with slip bands (Fig. 8). It was found that there is a brittle-to-ductile transition of the effect of $\sigma$ phase on alloys with increasing temperature, and that the transition temperature, above which the brittle influence of $\sigma$ phase on the alloy properties disappears, would be between about $600^\circ$ and $850^\circ$C, varying in some degree with the alloy composition (Fig. 9a). The hardness curves for the specially prepared pure $\sigma$ specimen (composition: Co 15%, Cr 40%, Mo 30%, Ni 15%) and $\gamma+\gamma'$ matrix (Fig. 9b) show that $\sigma$ phase has very high hardness at room and medium temperature, but that with increasing temperature it decreases faster than $\gamma+\gamma'$ phase. Between $800^\circ$C and $850^\circ$C the hardness values of both are nearly the same.

The harmful influence of $\sigma$ phase at elevated temperature is due to its weakening effects on al-
loysis when \( \sigma \) phase present in great quantities: decreasing the creep rupture life, enhancing the steady-state rate, and an earlier occurrence of third creep stage. Its precise mechanism needs further study.

**CONCLUSIONS**

\( \sigma \) phase in trace or small quantity has no substantial influence on the mechanical properties at room temperature and the stress-rupture life at \( 980^\circ C \), but decreases slightly the endurance life at \( 850^\circ C \). \( \sigma \) phase in large quantity and more leads to a considerable decrease of ultimate tensile strength, ductility, impact toughness and fracture toughness, but it has no marked influence on high frequency fatigue property at \( 700^\circ C \) and tensile property at elevated temperature. The stress-rupture life at \( 850^\circ C \) can be reduced to a very low level by large quantity of \( \sigma \) phase, and the elongation, also the steady state creep rate, increased considerably. The fracture characteristics are, intergranular on specimens without \( \sigma \) or with small amounts of \( \sigma \) after exposure and transgranular along the interface between \( \sigma \) and matrix on specimen with large quantities of \( \sigma \) phase.

At room and medium temperature, high dislocation density and stress concentration occur in the neighbourhood of the \( \sigma \) phase because it impedes the movement of dislocations. Even at rather small
deformation, cracking occurs along the \( \sigma \) plates and the material becomes brittle. At elevated temperature \( \sigma \) phase possesses some plasticity and becomes bent or zigzag under the interaction with slip bands. The hardness value of \( \sigma \) phase at room temperature is very high, but with increasing temperature it decreases faster than the \( \gamma + \gamma' \) matrix and above 850°C it is lower than that of \( \gamma + \gamma' \) matrix. Above the temperature of about 800-850°C the brittle influence of \( \sigma \) phase in large quantities on the properties of alloys disappears.

REFERENCES