SPECIFIC ASPECTS OF ISOTHERMAL AND ANISOTHERMAL FATIGUE OF THE MONOCRYSTALLINE NICKEL-BASE SUPERALLOY CMSX-6

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Abstract
An extensive study of the high-temperature isothermal and thermomechanical fatigue (TMF) behaviour of the "light" nickel-base superalloy CMSX-6 has been performed. Special emphasis was placed on a detailed microstructural interpretation of some specific aspects of the cyclic deformation behaviour. In the case of the isothermal fatigue tests, the main points of interest were the dependence on cyclic strain rate, effects of cyclic softening and directional coarsening, the cyclic stress-strain behaviour, mean stress and cyclic stress asymmetry effects and the dependence of the fatigue behaviour on different initial \( \gamma'/\gamma \) morphologies. In the TMF tests, the studies focused on the dependence of fatigue life on the strain-temperature cycle shapes, on directional coarsening effects for different cycle shapes, on the microstructural processes during a single cycle and on the effects of the strongly varying plastic strain rates within a cycle in a total-strain controlled test. A critical comparison between the isothermal and the TMF behaviour permits several conclusions to be drawn. In particular, it follows that while isothermal tests can provide a valuable guideline for the understanding of TMF, they are inadequate for a more detailed interpretation.

1. Introduction
Monocrystalline \( \gamma'/\gamma \)-hardened nickel-base superalloys, exhibiting superior high-temperature strength properties, are nowadays commonly used for advanced turbine blading of aircraft jet engines [1]. High-temperature creep strength and thermomechanical fatigue (TMF) resistance are considered to be the mechanical properties of major concern. While numerous detailed studies on the high-temperature creep behaviour of nickel-base superalloys have been performed in the past, cf. for example [2-7], much less work has been done on the isothermal [8-10] and, in particular, on the anisothermal TMF fatigue properties of monocrystalline superalloys, cf. [11-13]. Knowledge of the isothermal fatigue behaviour can provide a valuable guideline for the understanding of TMF but is, by itself, inadequate in order to draw satisfactorily with the much more complex deformation and damage processes occurring during TMF.

The goal of the present work is to gain a deeper understanding of the microstructural processes that govern high-temperature fatigue and, in particular, thermomechanical fatigue, of the "light" monocrystalline nickel-base superalloy CMSX-6. For that purpose, the studies focused on specific aspects of isothermal fatigue believed to be relevant also to TMF and on the material behaviour under selected well-defined TMF test conditions. The results of these investigations are contrasted against each other, and some general conclusions are drawn.

2. Experimental Procedure
In the present work monocrystalline rods of the \( \gamma'/\gamma \)-hardened nickel-base superalloy CMSX-6 (composition in wt.\%: 9.76 Cr, 3.01 Mo, 1.96 Ta, 4.70 Ti, 5.23 Co, 4.81 Al, bal. Ni) with orientations that lay within \( 10^\circ \) near [001] were used. These rods had been supplied by Thyssen-Güß AG, Feingußwerk Bochum, in the cast and heat-treated state. After machining and electropolishing, the fatigue test specimens had gauge lengths of 12 mm and a diameter of 9 mm. The microstructure consisted of fairly regularly arranged cuboidal \( \gamma' \) particles with 470 nm edge length, occupying a volume fraction of \( \geq 0.55 \). The constrained misfit parameter was determined by high-resolution X-ray diffraction as \( \delta \approx -10^{-3} \).

The fatigue tests were performed on uncoated specimens on a servohydraulic test machine (MTS 880) that had been equipped with high frequency induction coil heating and programming facilities for thermomechanical fatigue tests [14]. A high-vacuum chamber permitted tests in either air, arbitrary gaseous environments or in high vacuum. The isothermal and anisothermal fatigue tests in this study were all performed in closed-loop total strain control at prescribed total strain range \( \Delta e_T \). The corresponding plastic strains \( \varepsilon_{pl} \) could be calculated in all cases by taking into account the temperature dependent elastic strains, determined via the temperature-dependent Young's modulus. The latter had been measured previously in the testing machine.

The TMF-tests were performed with in-phase (IP), out-of-phase (OP) and clockwise and counterclockwise diamond (CD, CCD) temperature-total strain cycles. These cycle shapes are shown in plots of temperature \( T \) versus total strain \( e_t \) in fig. 1. In general, a total strain range of \( \Delta e_T = 10^{-2} \), an upper temperature of 1100 °C (or in some cases 900 °C) and a lower temperature of 600 °C were used. The cycle time was \( t_c = 300 \ s \), resulting in a total strain rate \( \dot{e}_t = 6.67 \cdot 10^{-5} \ s^{-1} \). The tests were always started at zero strain at the lowest possible temperature. For further details, see [15,16].

3. Experimental Results and Discussion

3.1 Isothermal Fatigue
In the following we report and discuss some noteworthy features of the isothermal fatigue behaviour.

3.1.1 Cyclic Deformation Behaviour Cyclic deformation curves obtained at different temperatures between 950 °C and 1100 °C and at \( \Delta e_T = 10^{-2} \), \( \dot{e}_t = 5 \cdot 10^{-3} \ s^{-1} \), are displayed in fig. 2 in the form of peak tensile and compressive stresses versus the num-

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ber of cycles $N$ (fig. 2a) and mean stress amplitude $\Delta \sigma/2$ and mean stress $\sigma_m$ versus $N$ (fig. 2b), respectively. As expected, the stress levels decrease with increasing temperature. At the same time, the fatigue lives decrease, mainly as a consequence of the increasing oxidation. An interesting effect is found with regard to the mean stresses $\sigma_m$ which develop during cyclic deformation. Up to 1050 °C, these mean stresses are compressive; at the highest temperature investigated (1100 °C), a tensile mean stress is measured. These results indicate a cyclic stress asymmetry which changes sign around 1075 °C. We return to stress asymmetry effects again in section 3.1.5.

3.1.2 Dependence of the Cyclic Deformation Behaviour on Cyclic Strain Rate The effect of cyclic strain rate on the cyclic deformation behaviour was studied at a temperature of 1100 °C at $\Delta \gamma = 10^{-2}$ for the total strain rates $\dot{\gamma}$ of $6.67 \cdot 10^{-4}$ s$^{-1}$ and $5 \cdot 10^{-3}$ s$^{-1}$. Figure 3 shows some corresponding hysteresis loops. Due to the enhanced strain-rate sensitivity at high temperatures, the peak stress at the higher strain rate was initially about 25 % higher than at the lower strain rate. In addition, very severe cyclic softening related to a "directional coarsening" of the $\gamma/\gamma'$ microstructure was observed at the lower but not at the higher strain rate, compare fig. 4a. As a consequence of these effects, the elastic strain amplitude was much larger at the higher strain rate and hence the plastic strain amplitude significantly smaller than at the lower strain rate. Accordingly, in the sense of a Manson-Coffin-type behaviour, fatigue life was

Figure 1: Temperature $T$ and total strain $\varepsilon_t$ versus time $t$ for different TMF cycle shapes. a) In-phase TMF (IP); b) Out-of-Phase TMF (OP); c) Clockwise diamond TMF (CD); d) Counterclockwise diamond TMF (CCD).

Figure 2: Isothermal cyclic deformation curves for different temperatures. a) Tensile and compressive peak stress $\sigma$ versus number of cycles, $N$; b) Mean stress amplitude $\Delta \sigma/2$ and mean stress $\sigma_m$ versus number of cycles, $N$.
3.1.3 Effects of "Directional" Coarsening at Low Cyclic Strain Rate

It is well known that coarsening of the \( \gamma / \gamma' \) microstructure can occur during deformation at sufficiently high temperature. In the case of the superalloy SRR 99, it has been shown [8,9] that, whereas during fatigue at 950 °C (\( \Delta \varepsilon_1 = 1.2 \cdot 10^{-2} \)) at a relatively high total strain rate (\( \dot{\varepsilon}_1 = 10^{-3} \text{ s}^{-1} \)), the coarsening effects are negligible, severe coarsening leading to an inclined raft-like \( \gamma / \gamma' \) structure, accompanied by pronounced cyclic softening and reduced fatigue life, occurs at a lower strain rate (\( \dot{\varepsilon}_1 = 10^{-5} \text{ s}^{-1} \)).

In our own studies at a rather high temperature (1100 °C), compare fig. 4, qualitatively similar results were obtained. Some coarsening of the initially cuboidal \( \gamma' \) precipitates (fig. 5a) was observed even after fatigue at the higher strain rate, with a tendency of raft formation perpendicular to the stress axis (fig. 5b), presumably in response to the positive mean stress. The changes in the \( \gamma / \gamma' \) microstructure after fatigue at the lower strain rate were more pronounced, both with regard to the amount of coarsening and with respect to the change of the morphology (fig. 5c). The edges of the coarsened \( \gamma' \) precipitates were found to lie more or less parallel to the traces of the \{111\} glide planes. Thus, "soft" \( \gamma \) channels had developed along the glide planes. In the related work of Portella et al. [8,9], these soft \( \gamma \)-channels were even more extended.

It is interesting to discuss to what extent this kind of directional coarsening is induced by annealing at the high temperature and/or the deformation process. In order to be able to perform the comparison for larger times, an additional test was conducted in vacuum at the higher strain rate, and the data were also used. The results shown in fig. 4a, plotted in the form of the cyclic stress amplitude against time (rather than against the number of cycles) for the two cyclic strain rates, compare fig. 4b, sheds some light on this question. As expected, the initial stress amplitude at the lower strain rate is lower than that at the higher strain rate. What seems surprising, is that the subsequent softening (decrease of stress amplitude as a function of time) is comparable for the two strain rates. In fact, the rate of softening is even somewhat larger for the higher strain rate. This suggests that, in both cases, the time of exposure to the high temperature is important in promoting the coarsening process, but that, in addition, the amount of strain accumulated during that time also supports the coarsening process, leading to a slightly larger softening rate at the higher strain rate.

3.1.4 Cyclic Stress–Strain Behaviour

The cyclic stress–strain behaviour, was studied in a multiple step test (\( 5 \cdot 10^{-3} < \Delta \varepsilon_l < 2.4 \cdot 10^{-2} \)) up to plastic strain amplitudes of about \( \Delta \varepsilon_{pl}/2 = 4 \cdot 10^{-3} \) at \( \dot{\varepsilon}_1 = 10^{-3} \text{ s}^{-1} \) at a temperature of 950 °C. A set of typical "saturated" hysteresis loops is shown in fig. 6. The cyclic stress–strain behaviour exhibits a significant stress asymmetry, the compressive saturation stresses being significantly larger at the higher than at the lower strain rate. We shall return to the coarsening effects in the next section.
higher than the tensile saturation stresses, in particular in the range $2 \cdot 10^{-3} < \Delta \varepsilon_p/2 < 3 \cdot 10^{-3}$ as shown in figs. 7 and 8. This stress asymmetry is in accord with that presented earlier in fig. 2b for a comparable temperature.

It should be noted that the hysteresis loops are slightly displaced to positive plastic strains, as shown in fig. 8 for the innermost loop. This is related to the fact that, in the first few cycles, more plastic strain is accumulated in tension than in compression, cf. fig. 9 and section 3.1.5. Since this plastic strain bias toward tension could distort the picture shown in fig. 8, the data were also evaluated, using the centre of the inner hysteresis loop as the origin. This procedure reduced the magnitude of the cyclic stress asymmetry somewhat but did not change the essence of the conclusions drawn earlier.

The shapes of the saturated hysteresis loops shown in fig. 5 differ in the tensile and compressive branches; the tensile branch exhibits a point of inflection in most cases. The same is true for the tension and compressive cyclic stress-strain curves (fig. 7). In this context, it appears appropriate to point out that, in monotonic tensile testing in the same temperature range, the stress-strain curve consists of several clearly distinguishable stages and also exhibits a point of inflection. Of course, this analogy must be confined to a small range of strain, comparable to that in cyclic deformation. In the case of the monotonic deformation,
the different stages of deformation were related to a sequence of deformation processes in the γ-channels perpendicular and parallel to the stress axis, respectively, and to cutting of the γ'/raft structures [17].

3.1.5 Cyclic Stress Asymmetry In this section we wish to discuss in a little more detail the effects of cyclic stress asymmetry and mean stress presented earlier (figs. 2, 4, 6, 7 and 8). Three findings are noteworthy with regard to total strain-controlled tests:

a) Below a temperature of about 1050 °C the cyclic stresses are larger in compression than in tension, leading to a negative mean stress.

b) Above that temperature, namely at 1100 °C in our work, the situation reverses: the tensile stresses now exceed the compressive stresses and a positive mean stress is found.

c) The tensile cyclic stress-strain curve below 1050 °C exhibits a point of inflection.

For the cyclic stress asymmetry below 1050 °C (\(\mu_m < 0\)), the cyclic deformation experiments at constant total strain range \(\Delta \varepsilon_1\) show that more plastic deformation occurs in the tensile half cycles of the first few cycles than in the compressive half cycles. Figure 9 shows an example for \(\Delta \varepsilon_1 = 10^{-5}, \dot{\varepsilon}_p = 5 \times 10^{-3} \text{ s}^{-1}\). Here the first three cycles are plotted in the form of stress \(\sigma\) against plastic strain \(\varepsilon_{pl}\). While the plastic strain reached in compression is virtually constant, the tensile plastic strain increases with continued cycling. As a consequence the tensile plastic strain and hence the tensile stress decrease, thus leading to an increased compressive mean stress, in agreement with the results shown in fig. 2. At present, the explanation of the physical origin of the different cyclic stress asymmetries observed below and above about 1050 °C, is not straightforward. TEM studies indicate that dislocation activity in cyclic deformation is confined to the soft γ-channels. Hence, an explanation must be sought in the mechanisms of dislocation motion in the γ-channels whose deformation is, however, constrained by the two-phase γ'/γ' morphology. For γ' cuboids, it is expected that, under the combined action of the external stress and the coherency stresses due to a negative lattice mismatch, deformation in the tensile and compressive half-cycles occurs preferentially in the γ-channels that lie perpendicular and parallel to the stress axis, respectively, compare [18]. Such considerations could possibly explain the fact that a point of inflection is found in the tensile but not in the compressive cyclic stress-strain curve. For other γ' raft morphologies, e.g. raft-like structures, geometric details such as the width of the γ-channels measured parallel to the glide planes would have to be taken into account. While corresponding detailed calculations are not available at present, it is felt that in order to explain the change of the cyclic stress asymmetry, some additional mechanism must be considered.

3.1.6 Effects of Prerafting on the Fatigue Behaviour Another point of interest concerns the effect of the γ'/γ' rafts, which are introduced perpendicular to the stress axis during high-temperature creep, on the fatigue behaviour. Such rafts are also found in turbine blades that have been subjected to service conditions [19]. Lupinci et al. [20] have investigated the effect of \(\gamma'/\gamma'\) rafts that were introduced by prior high-temperature creep on the high-temperature fatigue crack propagation. These authors showed that, for specimens with rafts lying perpendicular to the stress axis, the crack propagation was facilitated, compared to specimens containing cuboidal γ' precipitates. In order to test the effect of such γ'/γ' rafts introduced by high-temperature creep, Ott and Mughrabi [21] carried out isothermal fatigue tests (\(\Delta \varepsilon_1 = 0.9 \times 10^{-2}, \dot{\varepsilon}_p = 5 \times 10^{-3} \text{ s}^{-1}\) at 950 °C) in air on specimens having initially the cuboidal γ' particle morphology and on other specimens in which γ'/γ' raft structures which were either perpendicular or parallel to the stress axis had been introduced by a small (\(< 0.5\%\) prior tensile or compressive creep strain, respectively. These γ'/γ' microstructures are shown in fig. 10. Figure 11 shows some results of the fatigue tests. It was found that the fatigue life was enhanced, when the raft structure was parallel to the stress axis. In this case, a deflection of the crack into a direction along the raft structure was observed. On the other hand, for the raft structure perpendicular to the stress axis, fatigue life was reduced, and very smooth crack growth was facilitated perpendicular to the stress axis, as found also by Lupinci et al. [20]. In high vacuum, the fatigue lives were generally larger by a factor of about two. Otherwise, similar results were obtained.
Figure 10: Different γ/γ' microstructures of fatigue samples. a) Initial cuboidal γ' precipitates; b) γ/γ' raft structure parallel to stress axis after small compressive creep strain at $T = 1050 \, ^\circ\text{C}$; c) γ/γ' raft structure after small tensile creep strain at $T = 1050 \, ^\circ\text{C}$, γ' etching, the γ' phase appears dark.

Figure 11: Cyclic deformation curves at $T = 950 \, ^\circ\text{C}$, $\Delta \epsilon = 0.9 \cdot 10^{-3}$, $\epsilon_t = 0.9 \cdot 10^{-2} \, \text{s}^{-1}$ of specimens with the γ/γ' microstructures shown in fig. 10. Plots of mean stress amplitude $\Delta \sigma/2$ and mean stress $\sigma_m$ versus the number of cycles $N$.

3.2 Anisothermal Thermomechanical Fatigue

3.2.1 Cyclic Deformation, Fatigue Lives Figure 12 summarizes the TMF deformation behaviour for an upper temperature of 1100 °C in plots of the tensile and compressive peak stresses (fig. 12a) and the mean stress amplitude $\Delta \sigma/2$ versus the number of cycles (fig. 12b).

Out-of-phase (OP) TMF leads to the shortest fatigue lives, in-phase (IP) TMF to the longest; diamond cycles (CD, CCD) yield intermediate fatigue lives. The explanation is based on the fact that fatigue failure was found to occur predominantly by mechanical shear, favoured by large tensile stresses. In OP (IP) tests, a significant tensile (compressive) mean stress develops, and the tensile stresses are hence largest (smallest), the tensile peak stress being reached at the lower (upper) temperature. In the following, emphasis will be laid on the results of the OP TMF cycle shape which is considered to be the most damaging cycle shape in the present study.

3.2.2 Directional Coarsening, Effects on Fatigue Life In OP TMF tests with an upper temperature of 900 °C (lower temperature 600 °C) only a very small plastic strain amplitude developed. Failure occurred predominantly by local mechanical shear. SEM showed that the cuboidal γ' particle shape had been completely preserved. In all other TMF tests with upper temperatures of 1100 °C, directional coarsening was observed, leading to more or less well developed γ/γ' raft structures perpendicular (IP, CCD) or parallel (OP, CD) to the stress axis [22]. Examples of the γ/γ' raft structure found for the IP and the OP TMF tests are shown in fig. 13. The orientation of the rafts perpendicular (parallel) to the stress axis in the IP (OP) tests is as expected in the sense that, at the high temperature at which coarsening occurs, the specimen is under a tensile (compressive) stress.
It would, of course, be interesting to know whether these raft structures affect fatigue life and, if so, in what manner. For that purpose, specifically designed TMF tests would have to be performed on specimens with different well defined initial \(\gamma/\gamma'\)-microstructures. The observation of a particular \(\gamma/\gamma'\)-raft structure in a specimen that has exhibited an extended or reduced TMF life does not permit the conclusion that TMF life was enhanced or reduced by the formation of this particular \(\gamma/\gamma'\)-raft structure. Thus, Engler-Pinto Jr. et al. [23] found that, in SRR 99, specimens subjected to a so called thermal-fatigue-based (TFB) cycle, a well-developed raft structure was formed parallel to the stress axis and that the TFB fatigue life was larger than that observed after OP TMF or thermal fatigue. They concluded that a raft structure parallel to the stress axis seems to have a beneficial effect on the TMF life. On the other hand, the results of Kraft et al. obtained on the alloy CMSX-6 [21], as shown in figs. 12 and 13, show clearly that, in their work, fatigue life was shortest for the OP TMF test during which a raft structure developed parallel to the stress axis and longest for the IP TMF test which led to a raft structure perpendicular to the stress axis. In other words, these results simply show that the raft structures observed after anisothermal fatigue are a consequence of the type of anisothermal fatigue test performed but provide no unequivocal evidence on whether and how these raft structures affect anisothermal fatigue life. Thus, in the OP TMF tests discussed previously, any beneficial effect that the raft structure parallel to the stress axis may have had (compare, for example, the beneficial effect discussed earlier for isothermal fatigue), was obviously more than compensated by the damaging effect of the high tensile stress during the cold phase of the OP TMF cycle.

3.2.3 Microstructural Processes During a Single Cycle. Valuable insight into the processes during a single TMF cycle is gained by following the course of stress \(\sigma\), plastic strain \(\varepsilon_{pl}\), and temperature \(T\) versus time \(t\) during one cycle, see fig. 14. In TMF OP tests with an upper temperature of 900 °C, the shape of the stress-strain hysteresis loop is complex, and the plastic strain is barely measurable, as shown in fig. 14a, for cycles number 1, 100 and 500. Nonetheless, there is a cumulative accumulation of...
very small cyclic microplastic strains in the sense that, within 100 cycles, a permanent negative strain of about \(-1 \cdot 10^{-3}\) develops, accompanied by the build-up of a tensile mean stress of 150 MPa. No further changes are measurable up to 500 cycles. In TMF OP tests with an upper temperature of 1100 °C (fig. 14b), significant microplastic yielding in compression and the development of an appreciable tensile mean stress seem to occur in the hot phase during the first quarter cycle. After 100 cycles, the specimen has suffered a permanent negative plastic strain of about \(-3 \cdot 10^{-3}\) and has acquired a tensile mean stress of about 300 MPa. No significant further changes occur within the next 100 cycles.

3.2.4 Variation of Cyclic Plastic Strain Rate During a Cycle

We now discuss the changes of the plastic strain rate \(\dot{\epsilon}_{pl}\) within an OP TMF cycle. The corresponding hysteresis loop is shown in fig. 15a. For a triangular linear stress course, the \(\dot{\epsilon}_{pl}(t)\) course is strongly non-linear, compare fig. 15b. As a consequence, the instantaneous plastic strain rate \(\dot{\epsilon}_{pl}\) (slope of \(\dot{\epsilon}_{pl}\)) changes continuously, being close to zero most of the time and approaching, in the case of an OP TMF test, a significant value only during microyielding in the approach to the maximum temperature in the compression phase [14]. This variation of instantaneous plastic strain rate within a cycle is more severe than during isothermal fatigue. A detailed discussion of these effects has been given recently [14]. Some consequences are as follows. In a typical TMF test on a bulk specimen, the cycle time is in the order of several 100 seconds. Thus, the mean cyclic strain rate is very low, being significantly less than \(10^{-4}\) s\(^{-1}\) for the mean total strain rate \(\dot{\epsilon}_{t}\) and another one or two magnitudes lower for the mean plastic strain rate \(\dot{\epsilon}_{pl}\). As shown for the isothermal tests, compare figs. 4 and 5, the cyclic deformation behaviour for very low strain rates differs significantly from that at higher strain rates and leads to drastic coarsening and softening effects. Thus, a comparison of the cyclic deformation of TMF tests with data...
obtained in typical thermal fatigue tests on wedge-shaped specimens with much shorter cycle times is problematic. Further complications arise from the fact that the damage mechanisms and the process of crack propagation are quite different in the two cases, compare [24]. One step to improve the comparability would be to perform the TMF tests on hollow specimens in the hope to increase the strain rate by about one order of magnitude, compare [25]. Such work is in progress.

4. Conclusions

On the basis of the conducted experiments and, in particular, by a comparison of the results observed for isothermal and for anisothermal thermomechanical fatigue, several conclusions can be drawn. Here we note:

a) There are fundamental differences between isothermal and anisothermal fatigue behaviour. Hence, any similarities must be viewed critically.

b) Directional coarsening of the γ/γ' microstructure is qualitatively different in isothermal and anisothermal fatigue, and even for similar γ/γ' raft structures accompanying changes of fatigue lives can be quite different. The microstructural effects seem more important in isothermal than in anisothermal fatigue.

c) At high temperatures, drastic cyclic softening, caused by marked coarsening of the γ/γ' microstructure, occurs in particular at low strain rates.

d) In total strain controlled tests, the instantaneous plastic strain rate varies in a complex manner during a single cycle. Because of the high strain rate sensitivity at high temperatures, the consequences of such behaviour deserve more attention.

e) Mean stresses must be considered in both isothermal and anisothermal fatigue, being more important in the latter case.

f) In TMF tests, the temperature changes simultaneously with the mechanical strain. As a consequence, the effects of strain rate are more pronounced than in isothermal tests.

g) In TMF tests on bulk specimens, the cycle times are in the order of minutes, and the (plastic) strain rate is according-ly much lower than in thermal fatigue tests with typical cycle times in the order of seconds. In addition, the nature of fatigue damage is different in both cases. Thus, a direct comparison is problematic.

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