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High temperature thermal diffusivity of nickel-based superalloys and intermetallic compounds

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ABSTRACT

By means of an installation developed in our laboratory, we measured the thermal diffusivity (α) as a function of temperature for several single and polycrystal nickel-based superalloys as well as for different intermetallic compounds with a L1₂ (Ni₃Al, Ni₃Si, Ni₃Ge, Ni₃Fe, Zr₃Al, Co₃Ti), L1₀ (TiAl) or B2 (NiAl) structure. In the case of nickel-based superalloys, the experiments pointed out an unexpected but reproducible slope change in the $\alpha=f(T)$ curves at about 750°C, which is not explained at this time. It was shown that the initial microstructure, the crystal orientation and the grain size seem to have no noticeable influence on the thermal behaviour. Slight variations between different alloys were rather attributed to a chemical composition effect. In the case of intermetallic compounds the temperature dependence of the thermal diffusivity appeared to drastically change from an alloy to another. Thus, no clear relation could be found between thermal behaviour, crystallographic structure and strength temperature dependence of these alloys.

INTRODUCTION

The thermal diffusivity of a material is defined as $\alpha = \lambda / \rho c$ where λ , ρ and c are the thermal conductivity, density and specific heat, respectively. From a thermal point of view, it characterizes the ability of the material to diffuse a thermal perturbation. From the material side and due to this definition, the measurement of this property can reveal processes related to chemical composition as well as structural transformations.

The most commonly used technique for measuring thermal diffusivity is called "flash method". It consists in applying a pulse of energy on the front face of a plate sample and in following the resulting thermal evolution of its rear face. Using an apparatus based on this principle, we recently performed experiments on single crystals of nickel-based superalloys and of a Ni₃Al intermetallic compound [1]. They revealed an unexpected thermal behaviour since the $\alpha=f(T)$ curves of superalloys and Ni₃Al presented a sudden slope change and a maximum, respectively, at about 750°C. This phenomenon was not explained although the analogy with mechanical behaviour (end of a plateau or maximum of yield stress at the same temperature) was noted. In the meantime, new experiments have been performed on different single and polycrystal superalloys with high volume fractions of γ' phase as well as on several intermetallic alloys. The aim of this study was to confirm our first results and to determine if the thermal-mechanical analogy was fortuitous or not. This paper reports and discusses the results of this investigation.

EXPERIMENTAL PROCEDURE

The chemical compositions of the six nickel-based superalloys concerned with the present work are given in Table 1, together with their initial heat treatments and their grain structure. The value of high temperature γ - γ' misfit is also indicated when available. As described in [1], TT1 and TT2 heat treatments applied to CMSX2 alloy resulted in coherent and "degenerated" precipitates, respectively. In the case of 01

polycrystal alloy, TT1 and TT2 treatments led to different grain sizes, as indicated in Table 1. Different intermetallic compounds, the chemical compositions of which are reported in Table 2, were also studied. The structure of the corresponding ordered phase as well as the temperature of the peak of maximum strength if any are also given. The polycrystalline alloys were elaborated using induction melting in a cold Cu crucible then submitted to homogenization heat treatments indicated in Table 2. As far as possible, these heat treatments were chosen similar to those used by Wee et al in their studies dealing with the temperature dependence of strength of L1₂ intermetallics [2,3].

Cylindrical samples ($\varnothing$ 10 mm, thickness 2 mm) were prepared by spark erosion and/or diamond saw cutting. Their main faces were polished to ensure a good parallelism, then sand-blasted. The installation used for the diffusivity measurements has been described elsewhere [4]. The pulse of energy is obtained with a laser YAG. The pulse duration is about 500 ms and the energy level ranges between 5 and 30 J. The thermal signal is detected with a HgCdTe detector cooled to liquid N₂ temperature. The sample is located in a furnace under neutral atmosphere. The measurement can be performed between room temperature up to 1500 °C with a minimum temperature step of 20 °C and with a precision better than 5%. The installation is completely controlled with a Macintosh II computer from APPLE. The software (developed in the LSG2M) is in charge of all the operations and allows automatic measurements on heating, cooling (5°C/mn) and versus time at any given temperature. As an example, a complete $\alpha=f(T)$ curve between 20 and 1400°C is obtained in 8 hours without any manipulation.

	Ni	Al	Co	Cr	Ta	W	Mo	Ti	δ (%)	grain orientat.	Heat Treatments
AM1	base	5.1	6.5	7.4	8.0	5.3	2.0	1.2	-0.25	<001> <111> D.S. <001> polycrystal	1300°C/3h. - air cooled
AM3	base	5.86	5.61	8.04	3.43	4.91	2.2	2.0	-	<001>	1310°C/3h. - a. c.
MC2	base	4.87	5.08	7.86	6.05	7.85	2.07	1.53	-0.4	<001>	1300°C/3h. - a. c.
01 alloy	base	5.0	-	6.0	7.0	-	2.0	-	+0.37	polycrystal	TT1: as cast (fine grains # 60 μ m) TT2: 1300°C/3h (coarse g.# 400 μ m)
CMSX2	base	5.6	4.6	8.0	6.0	8.0	0.6	1.0	-0.18	<001>	TT1: 1315°C/3h. - a. c. TT2: TT1 + cooling at -0.01°C/min
CMSX2'	base	5.3	5.2	7.4	7.7	7.3	0.5	0.2	-0.09	<001>	1315°C/3h. - a. c.

TABLE 1 : Chemical compositions (wt%), high temperature γ - γ' lattice mismatch δ (measured at 1050 °C or 1100°C ; ref. [5,6]), crystal orientations and initial heat treatments of the six superalloys studied in the present work

Alloy	Composition	Heat Treatment	ordered struct.	strength peak
Zr ₃ Al	25 at.% Al	1300°C/1h + 600°C/15 d furnace cooled	L1 ₂	# 500°C
NiAl	50 at.% Al	1000°C /4 d - f. c.	B2	no
Ni ₃ Ge	25 at.% Ge	1000°C /5 d - f. c.	L1 ₂	# 450°C
Ni-Ge	23.5 at.% Ge	1000°C /5 d - f. c.	L1 ₂	-
Ni ₃ Fe	25 at.% Fe	500°C /12 d - f. c.	L1 ₂	no
Co ₃ Ti	25 at.% Ti	1050°C /10 d + 1000°C /2d - f. c.	L1 ₂	# 750°C
TiAl	48.8 at.% Al + 2.2 at.% V	TT1 : 1400°C /0.5 h - f. c. TT2 : 1250°C /1 h - f. c.	L1 ₀	# 600°C
Ni ₃ Al single X	22.5 at.% Al + 0.25 at.% Hf	1200°C /1 d	L1 ₂	# 650°C
Ni ₃ Al	25 at.% Al	1200°C /4 d - f. c.	L1 ₂	-
Ni ₃ Si	25 at.% Si	1050°C/1 d + 900°C/2 d + 800°C/4 d - f. c.	L1 ₂	# 400°C

TABLE 2 : Chemical compositions and homogenization heat treatments of the intermetallic compounds studied in the present work. All samples were polycrystals except one Ni₃Al single crystal. The crystallographic structure of the corresponding ordered phase is also indicated together with the strength peak temperature in the case of a positive temperature dependence.

RESULTS AND CONCLUSIONS

Figure 1 shows the evolution of thermal diffusivity from room temperature up to 1300°C for the six nickel based superalloys with different orientations and different heat treatments. Table 3 gives the values of diffusivity measured at room temperature in the case of AM1 and 01 alloys with different grain structures (at least 20 measurements per sample). This set of experiments leads to the following conclusions :

- all the $\alpha=f(T)$ curves are very similar, confirming the existence of a clear change in the thermal behaviour of these alloys between 700°C and 800°C.
- heat treatments (i.e. microstructure and/or internal stress state), crystal orientation and grain size do not seem to explicitly influence the thermal diffusivity, at least in the limit of our experimental conditions. In the same way, no direct relation can neither be found between diffusivity and misfit magnitude.
- small variations can still be observed between the diffusivity levels of the different alloys. At the light of the preceding remarks, this is supposed to be mainly related to chemical composition variations.
- at higher temperatures, the slope of $\alpha=f(T)$ curve varies from an alloy to another (this is especially noticeable in the case of 01 alloy). The reason for this is not clear and works have to be done to precise the parameters responsible for these differences (precipitate volume fraction, dissolution kinetics, chemical composition ...).

Figure 2 shows the $\alpha=f(T)$ curves obtained for the intermetallic compounds of Table 2. The thermal evolution appears to drastically change from an alloy to another. It is verified that thermal diffusivity is influenced by slight variations of chemical composition (see for instance the case of Ni-Ge

FIGURE 1 : Temperature dependence of thermal diffusivity for the six superalloys of Table 1

AM1 alloy	single crystal	direct. solidified	polycrystal
	2.37 $\pm$ 0.03	2.40 $\pm$ 0.015	2.38 $\pm$ 0.01
01 alloy	fine grains (TT1 treatment)		coarse grains (TT2 treat.)
	2.81 $\pm$ 0.015		2.79 $\pm$ 0.01

TABLE 3 : Thermal diffusivity (mm²/s) measured at room temperature in the case of AM1 and 01 alloys with different grain structures (at least 20 measurements per sample)

alloys) and that it is very sensitive to magnetic transformation (Ni_3Fe) and probably also to order-disorder transformation (stoichiometric Ni_3Al and Ni_3Ge alloys). However, although some curves seem to present a singularity in the vicinity of the strength peak temperature (Ni_3Al , TiAl , Ni_3Ge), this set of experiments showed that this situation is not general. Moreover, no direct relation can even be found between the crystallographic structure and the general trend of the diffusivity temperature dependence. At this time no theoretical approach is still capable to forecast the thermal properties of this type of alloys. Thus, a more precise and systematic experimental characterization of their thermal behaviour appears to be necessary.

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FIGURE 9: Temperature dependence of thermal diffusivity for the different intermetallic compounds of Table 1. Strength peak temperatures are marked with an arrow.