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DIFFUSION NIOBIUM - NICKEL

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Abstract: *To study the inter-diffusion Nb / Ni, were created couples of Ni 99.98% Ni and Nb 99.98% which were placed in ampoules sealed under controlled atmosphere (Ar). These ampoules of quartz sealed are then placed in an oven for different time-temperature. The chosen treatment temperature is 1050 C. The times chosen were 7, 15 and 30 days. The pairs of inter-diffusion has been characterized by SEM (Scanning Electron Microscope), EDX analysis (Energy Dispersive X-ray Spectrometry) and by SIMS (Secondary Ion Mass Spectroscopy). For the first samples of 7 and 15 days, it is difficult to assess the exact nature of phase relative to the distant between the two interfaces. It is therefore necessary to conduct more thorough characterizations. Only for the sample maintained 30 days was 1050ºC, the concentration profile present levels that may be associated with the component Ni3Nb. Understand and control these mechanisms of diffusions would thus provide solutions to industrial problems (improved resistance to stress corrosion for example).*

Keywords: diffusion, niobium, nickel, SEM, EDX, SIMS

INTRODUCTION

A pure metal has physicochemical characteristics often not suited to industrial uses, is the solution to other chemical elements can significantly change the material properties, to "harden" this metal and thus increase its mechanical properties. Furthermore, these additions can also change the chemical properties of pure metal (in particular, their corrosion behavior) or even modify other features (ease of implementation for example).
The choice of nickel has been done because it serves as the base metal in many alloys such as, Ni-based super alloys that exhibit excellent corrosion resistance at high temperature dry and very good mechanical properties (tensile strength high creep resistance). These are materials of choice for jet engines (aerospace). They are also currently used in the nuclear field. In these alloys, the Ni is combined with alloying elements such as Cr, Fe, Al, Co, Nb and forming a solid solution called γ. Among these alloying elements, niobium has been chosen because it participates actively in building mechanical properties. First it hardens the material upon its entry into solid solution and secondly it may precipitate with nickel as a hardening phase γ'' (Ni3Nb). These precipitates are primarily responsible for the increase of mechanical properties of many superalloys. They improve the mechanical properties by slowing / blocking the movement of dislocations [1].
Niobium is an essential element of addition in these superalloys, or control the process of diffusion of niobium in nickel is an inherent basis for understanding the mechanisms of solidification and hardening in these alloys. Knowledge of the diffusion behavior of niobium in nickel-based alloys could lead eventually to gain more control over the distribution and diffusion of refractory elements.

EXPERIMENTAL DETAILS

In order to study the inter-diffusion Nb / Ni, the first step is to design the diffusion couples. Materials used: a bar of 99.98% Ni with a diameter of 12.2 mm and a tube of Nb 99.98% Nb with the outside diameter 20mm and the insider diameter of 12.2 mm. To create the pairs of Ni / Nb was used the precision cutting machine. To have the least impurity, the two materials must be carefully polished by mechanical polishing and electrochemical polishing.

After mechanical polishing the samples of nickel and niobium, they were subjected to electrochemical polishing. For each sample we used different mixtures. For samples of nickel we used a mixture of perchloric acid and acetic acid (2/3- 1 / 3 by volume), with an applied voltage of 70 V for an exposure time of 5 seconds. And niobium we used a mixture of 90% sulfuric acid and hydrofluoric acid 10%, with a time of 45 seconds and a voltage of 6V.[8] Once the surfaces were clean a mechanical press was used to insert the nickel in the cylinder of niobium. The couple was then placed in quartz ampoules sealed under controlled atmosphere (Ar) in an oven for different time-temperature couples. The times chosen were 7, 15 and 30 days.

The pairs of inter-diffusion has been characterized by SEM (Scanning Electron Microscope) to study the evolution of microstructure, EDX analysis (energy dispersive X-ray Spectrometry) was conducted to obtain profiles concentrations in surface and transverse section and by SIMS (Secondary Ion Mass Spectroscopy) was used to map the chemical elements in the areas of inter-diffusion and thus to directly study the distribution.

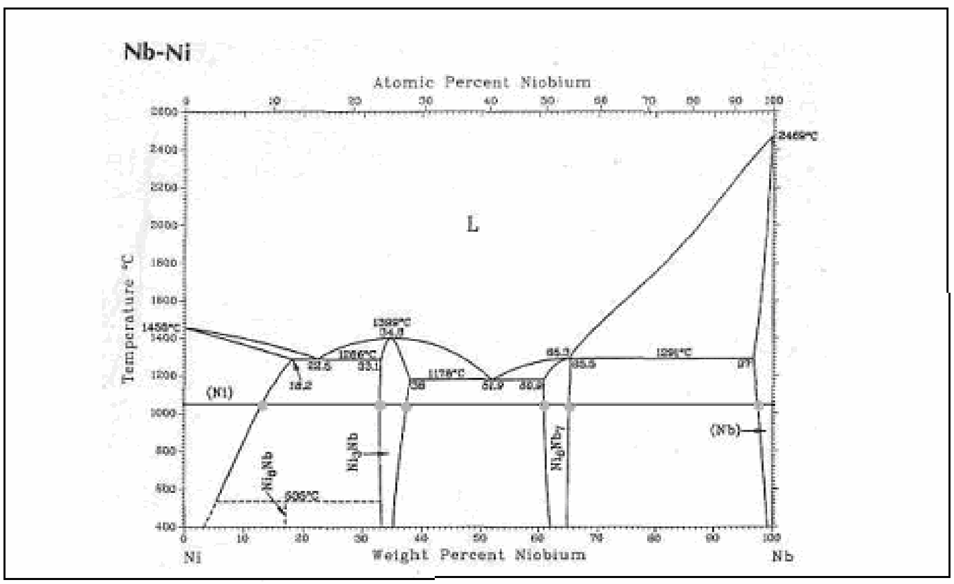


Fig.1 Phase diagram niobium-nickel [4,9]

Both parts of the couple was prepared separately by polishing on sandpaper on a machine Buelher Metaserver, followed by polishing a 1μm diamond paste up to a machine Trieffus 68201 and 83201. After that the couples were coated in resin. The evolutions of microstructure were observed with the Scanning microscopy type JEOL 6400.

RESULTS AND DISCUSIONS

A micrograph of the diffusion zone obtained after the sample of Ni-Nb was maintained in the oven for 7 days at 1050ºC. Given the variation in concentration in the area of diffusion, the phases present in the equilibrium diagram Ni-Nb may be present. For a better identification of these phases it was necessary to perform a more detailed analysis on the area of diffusion.

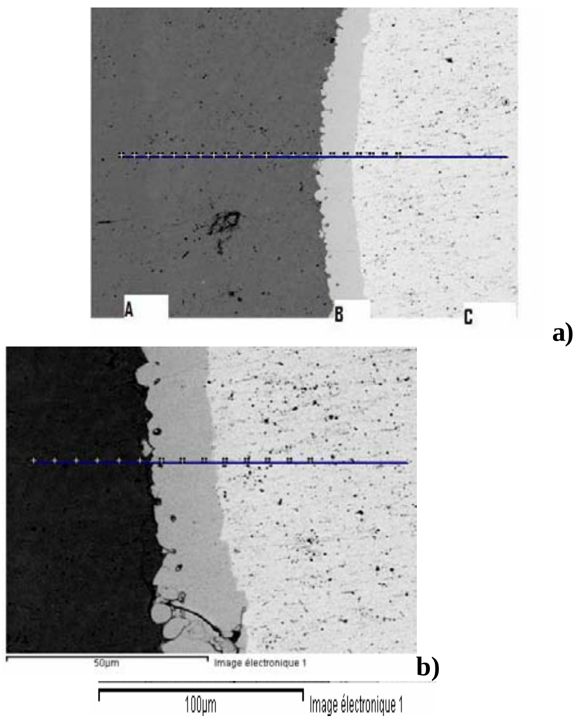
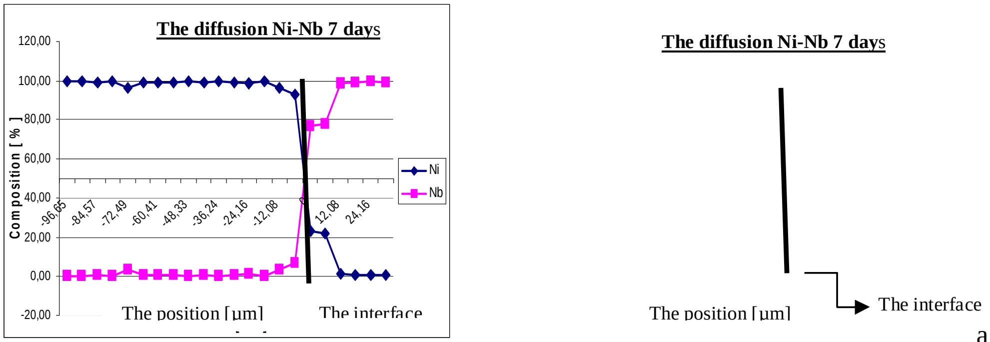
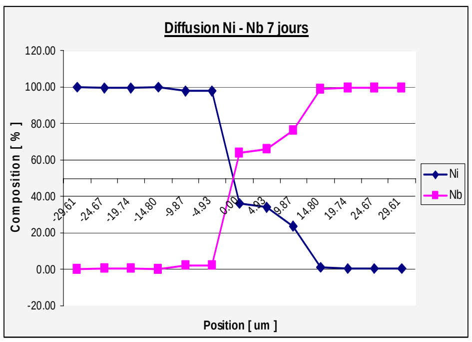


Fig. 2 Diffusion of niobium - nickel, dwells 7 days in the oven at 1050ºC, SEM a) A) Ni ; B) Zone Inter-diffusion ; C) Nb ; b) area of interest realize on the same couple



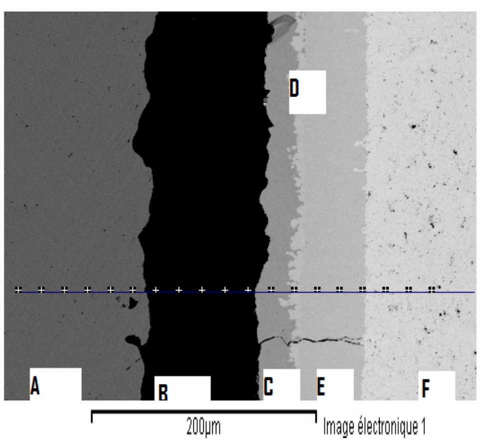
a)



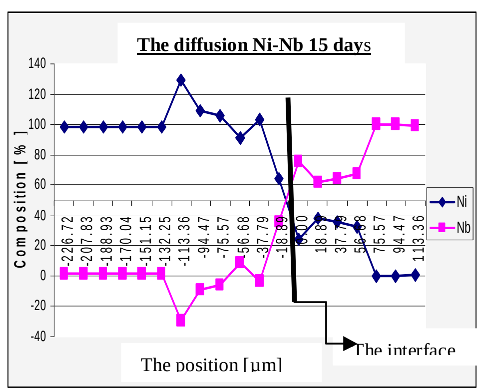
b)

Fig. 3 a) Concentration profile in the area of inter-diffusion for the sample of 7 days (wt%); b) Concentration profile in the area of inter-diffusion for the sample of 7 days (wt%)(details)

For the sample of 15 days the results are the following:



a)



b)

Fig. 4 Experimental results on the sample of Ni-Nb maintained in the oven for 15 days at 1050°C; a) A)Nickel, B)Resin, C),D),E) Zone d'inter-diffusion, F)Niobium ; b) Concentration profile in the area of inter-diffusion for the sample of 15 days (wt%)

A more detailed SEM analysis was performed because the sample was too thin and has allowed penetration of resin in it. Since the resin occupies an important place the image of the SEM was cut in two parts, one part for niobium and another for nickel.

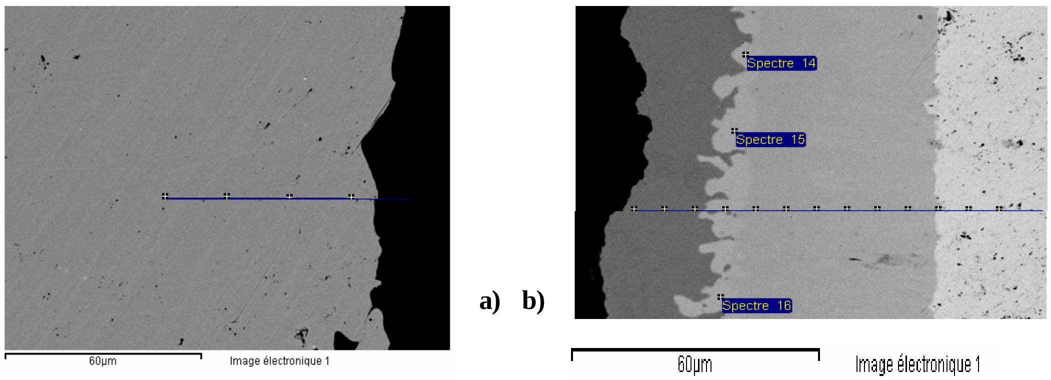


Fig. 5 Experimental results on the sample of Ni-Nb maintained in the oven for 15 days at 1050ºC; a) side nickel; b) side niobium.

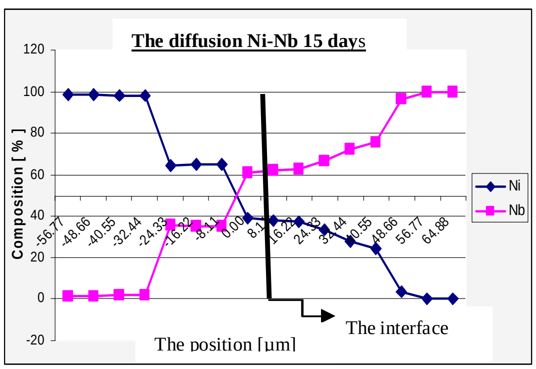


Fig. 6 Experimental results on the sample of Ni-Nb maintained in the oven for 15 days at 1050ºC; Concentration profile in the area of inter-diffusion for the sample of 15 days (wt%)

For the sample of 15 days, a SIMS analysis was performed at the interface between the niobium and the area of inter-diffusion.

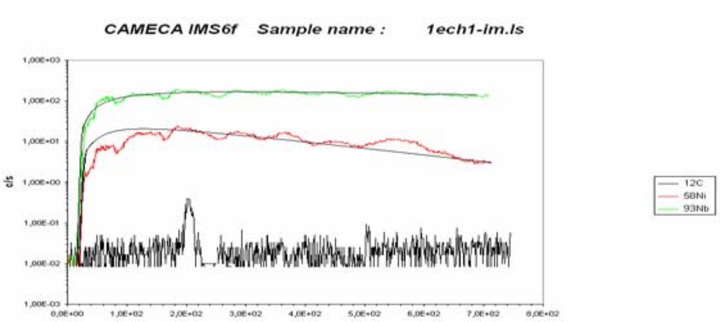


Fig.7 Experimental results on the sample of Ni-Nb maintained in the oven for 15 days at

1050ºC by SIMS analysis;

It is observed that the concentration of Ni down further we penetrate into the niobium.
For the sample the couple nickel-niobium maintained in the oven for 30 days at 1050ºC the results are:

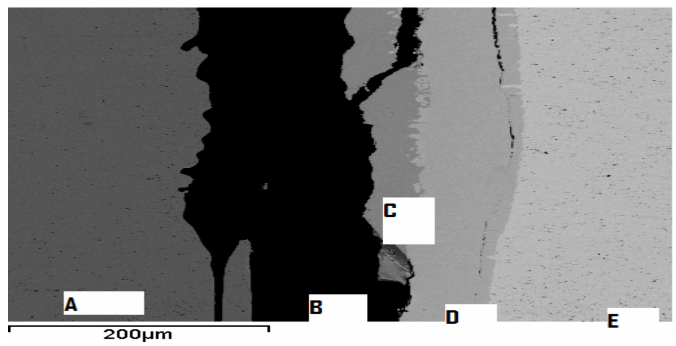
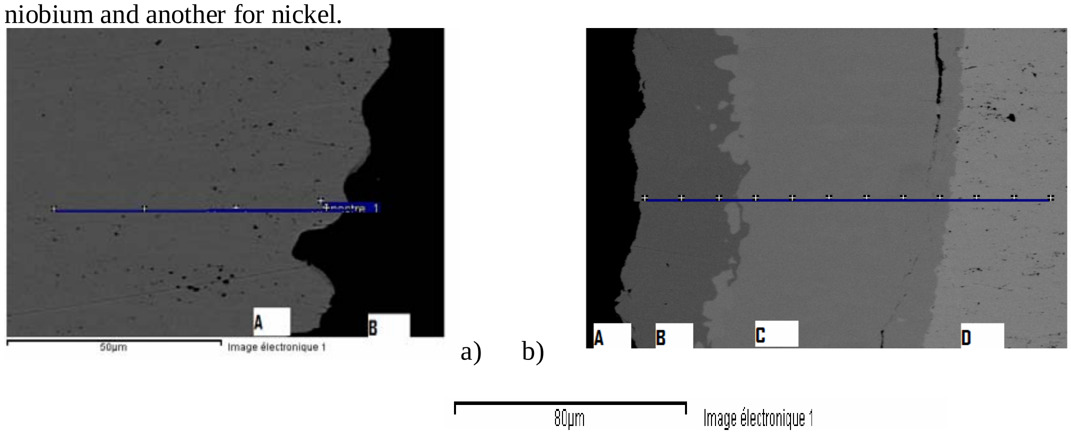


Fig. 8 Experimental results on the sample of Ni-Nb maintained in the oven for 30 days at 1050ºC; a) A)Nickel, B)Resin, C),D) Zone d'inter-diffusion, E)Niobium ;

Also for the experiment of 30 days the image of the SEM was cut in two parts, one part for niobium and another for nickel.



a) b)

Fig. 9 Experimental results on the sample of Ni-Nb maintained in the oven for 15 days at 1050ºC; a) side nickel; b) side niobium.

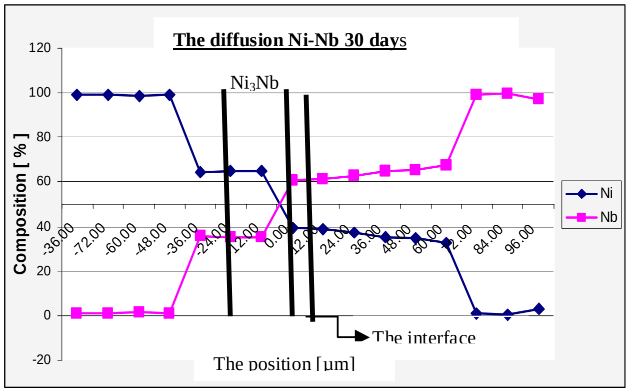


Fig. 10 Experimental results on the sample of Ni-Nb maintained in the oven for 30 days at 1050°C; Concentration profile in the area of inter-diffusion for the sample of 30 days (wt%)

In the graph of the concentration rofile for the sample of 30 days the area of Ni_3Nb can be observed.

CONCLUSIONS

The microscopic analysis with EDX of the samples shows for all samples the characteristic zones for base metals (nickel and niobium) and an area of inter-diffusion distribution.
The width of the inter-diffusion zone increases with the holding of the couple at the oven at 1050C a longer period of time.
For the first two samples, it is difficult to assess the exact nature of phase relative to the distant between the two interfaces. It is therefore necessary to conduct more thorough characterizations.
Only for the sample maintained 30 days was 1050C, the concentration profile present levels that may be associated with the component Ni3Nb.
Understand and control these mechanisms of diffusions would thus provide solutions to industrial problems (improved resistance to stress corrosion for example).

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