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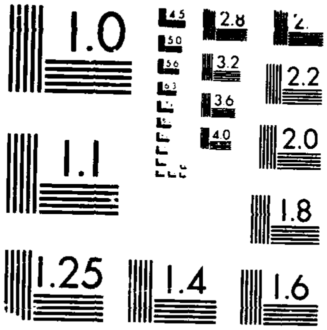

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ABSTRACT

Among the problems associated with long-term high temperature service of hybrid material systems is interdiffusional compatibility of the system's component materials. Efforts in this program have focussed on several area of this problem. One of the areas being examined are alkali metal and alkali earth metal diffusion barriers. These barriers are being applied by two different methods: ion implantation and a sol suspension slurry technique. For both methods, the system being utilized as a model is the W / Ni system. Preliminary results of barrier effectiveness are given. Also being studied are the interdiffusional kinetics of metal/intermetallic and intermetallic/intermetallic ternary systems. Hybrids being examined include W/Ni_3Al and TiAl/Ni_3Al.

KEYWORDS: NICKEL ALUMINIDES, TITANIUM ALUMINIDES.

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I. INTRODUCTION AND BACKGROUND

In many structural applications, conventional alloys or materials can no longer fulfill the increasing drastic service demands and/or long-term reliability requirements of many of the anticipated designs. High temperature material systems for Air Force jet engine applications are especially prone to increasingly rigorous heat resistant and life-cycle demands. In some respects the demands are a result of new designs that will improve product performance. For example, higher operating temperatures of jet and rocketry engines increase the thermodynamic efficiency and power output, while lowering both fuel consumption and exhaust emissions.

At present gamma/gamma prime strengthened superalloy single crystals place a ceiling on the operating turbine blade skin temperature of jet engines at approximately $1050^{o}C$ [1,2,3]. The need to push this operating temperature limit higher and also increase the material reliability at current operating temperatures has resulted in research attention redirected from conventional alloys to more novel materials concepts like structural ceramics, carbon-carbon systems, and such hybrid materials systems as oxide dispersion strengthened alloys and fiber reinforced alloys and/or reinforced intermetallics and ceramics. This program addresses hybrid materials systems for prolonged high temperature applications.

Hybrid materials systems intended for ambient temperature or mid-range temperature service require a certain degree of mechanical property compatibility between the system components. For example, metal/metal or graphite reinforced epoxy composites require ductile matrix behavior in order to ensure adequate load transfer from the matrix to the fiber components. For hybrid material systems intended for repeated thermal excursions to high temperatures, thermal-mechanical or thermal fatigue compatibility is also required. The situation becomes even more complex for hybrid systems that are subjected to prolonged high temperature excursions. In addition to mechanical and thermal-mechanical compatibility, time-reliable hybrid materials system components must also exhibit chemical compatibility at the intended service temperatures.

Thermal-mechanical compatibility is influenced by the thermal expansion among the composite components, the thermal conductivity of the hybrid components, and the ability of the components to accommodate strains associated with thermal cycling. The high temperature chemical compatibility is related to such thermally activated processes as interdiffusion and interfacial reaction kinetics, if any.

In general, the thermal-mechanical compatibility of hybrid and composite systems is well understood. For example, many researchers have used elasto-plastic analysis to describe and/or measure the effect of thermal mismatch in hybrid and composite systems [4-8].

A difference in the chemical potential between hybrid components can result in interdiffusion among the components provided sufficient thermal activation exists. High temperature chemical compatibility becomes increasingly important for higher operating temperatures for prolonged exposure times as a result of the time and temperature dependence of the kinetics of interdiffusion. Interdiffusion related interactions between hybrid components can affect the strengthening microstructures of the hybrid system, including the growth of tertiary reaction and potentially brittle phases at the component interfaces. Such interactions could result in the degradation of the mechanical properties of the hybrid materials system with time, resulting in time limited service constraints.

Current theoretical or analytical models are in the rudimentary stages with respect to predicting phase stability in complex systems and in forecasting the degree of interdiffusion and its effects. As a result predictive chemical compatibility between hybrid components can only be made on a restrictive basis. Actual confirmation of the compatibility and the amount of change has to be made experimentally.

Interdiffusion controlled reactions in high temperature hybrid materials systems have been reported in the literature with respect to such constitutionally simpler (metal/metal) hybrid systems as the tungsten fiber reinforced superalloys (TFRS) [1,9-30]. Much of this literature has Russian origins. The current state of understanding can be compacted as follows: (1) W-fiber and matrix recrystallization due to the interdiffusion, (2) W-fiber dissolution, and (3) intermetallic phase growth at the fiber/matrix interface. In addition, it should be noted that even in systems involving alloys strengthened by the so-called inert oxides, the temperature ceiling of use is controlled by oxide coarsening [31] and by the formation of voids resulting from unbalanced interdiffusion [32].

II. CURRENT STATUS

Our Research efforts during the initial 14 month period of this grant have been focussed on two separate areas of composite interface technology. One phase of the current research is devoted to the investigation of diffusion barrier coatings which will inhibit interdiffusion of reinforcing and matrix materials across fiber/matrix interfaces. The second, parallel effort is evaluating the interdiffusional stability and chemical compatibility of selected metal/intermetallic and intermetallic/intermetallic composite systems.

In response to the problem on interfacial compatibility of composite constituents we are investigating the effectiveness of several diffusion barrier layers which show promise. In order to test the feasibility of these barrier layers we have chosen the simple W / Ni system as a model. This system was chosen due to the well understood interdiffusional phase stability and kinetics of interdiffusion of the pair. The barriers which we are primarily interested in are alkali metal ions. Alkali ions being considered are Na, Ca, Ba, and K. These alkali and alkali earth metals possess very large molar volumes. See Table 1. It might be expected that by incorporating these atom in a thin layer at or near the interface that diffusional behavior may be modified due to the internal strain caused by these atoms in the lattice. However, if excessive strain is caused the lattice may recover through thermally activated processes and the additions may be ineffectual or even intensify interdiffusion. Diffusion couples incorporating these layers to evaluate their feasibility are being fabricated by two different methods.

TABLE 1

Elemental Molar Volumes

Ni	6.6 cm3
W	9.5 cm^3
Na	29.9 cm^3
Ca	32.7 cm^3
Ba	39.0 cm^3
K	45.0 cm^3

One method being employed for fabricating these alkali ion diffusion barriers is ion implantation. Diffusion couples have been made by ion implanting alkali ions into a tungsten substrate and subsequently ion beam sputtering a film of nickel over the implanted surface. Conditions which were utilized for this process are shown in Table 2. The nickel films were found to exhibit the typical sputtered microstructure of a thin epitaxial layer, followed by a region of fine equiaxed grains, with the remainder being long columnar grains oriented in the growth direction. See Figure 1. These diffusion couples were annealed at 1500K in vacuum for 50 hours. Composition--position plots were obtained for the couples using a calibrated EDS microprobe. Concentrations were determined at 1 micron intervals across the interdiffusion zone. An example of such a plot is shown in Figure 2. Due to the rather long time required to deposit the nickel film on the tungsten substrate, combined with the nickel's microstructure, a large degree of grain boundary diffusion is evident. This consequence of the relatively simple fabrication technique employed limits the analysis of these couples to a comparative analysis of the extent of interdiffusion between the different barriers employed as well as the control specimen without any barrier. Figures 3 and 4 illustrate the effect of the individual barrier atoms relative to the couple without any barrier and the couple initial condition. As can be seen, calcium atoms were found to have a pronounced effect on reducing interdiffusion in the W/Ni system while sodium was found to have a negligible effect the interdiffusional penetration. In fact, preliminary data suggests that potassium may even promote

TABLE 2

Ion Implantation Conditions	
Ion Density	6.4E-11 cm^{-2}
Accelerating Potential	190 kV
Median Penetration	250-350 nm

Ion Beam Sputtering Conditions	
Substrate Temperature	400°C
Deposition Time	70 Hours

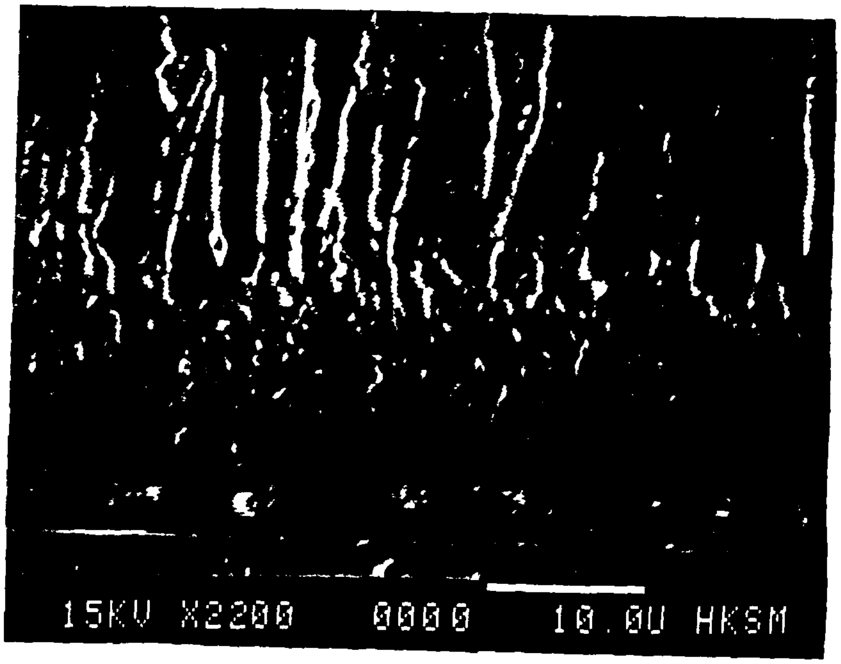


Figure 1. As-Received Microstructure of Ion Beam Sputter Deposited Ni Films.

Figure 2.

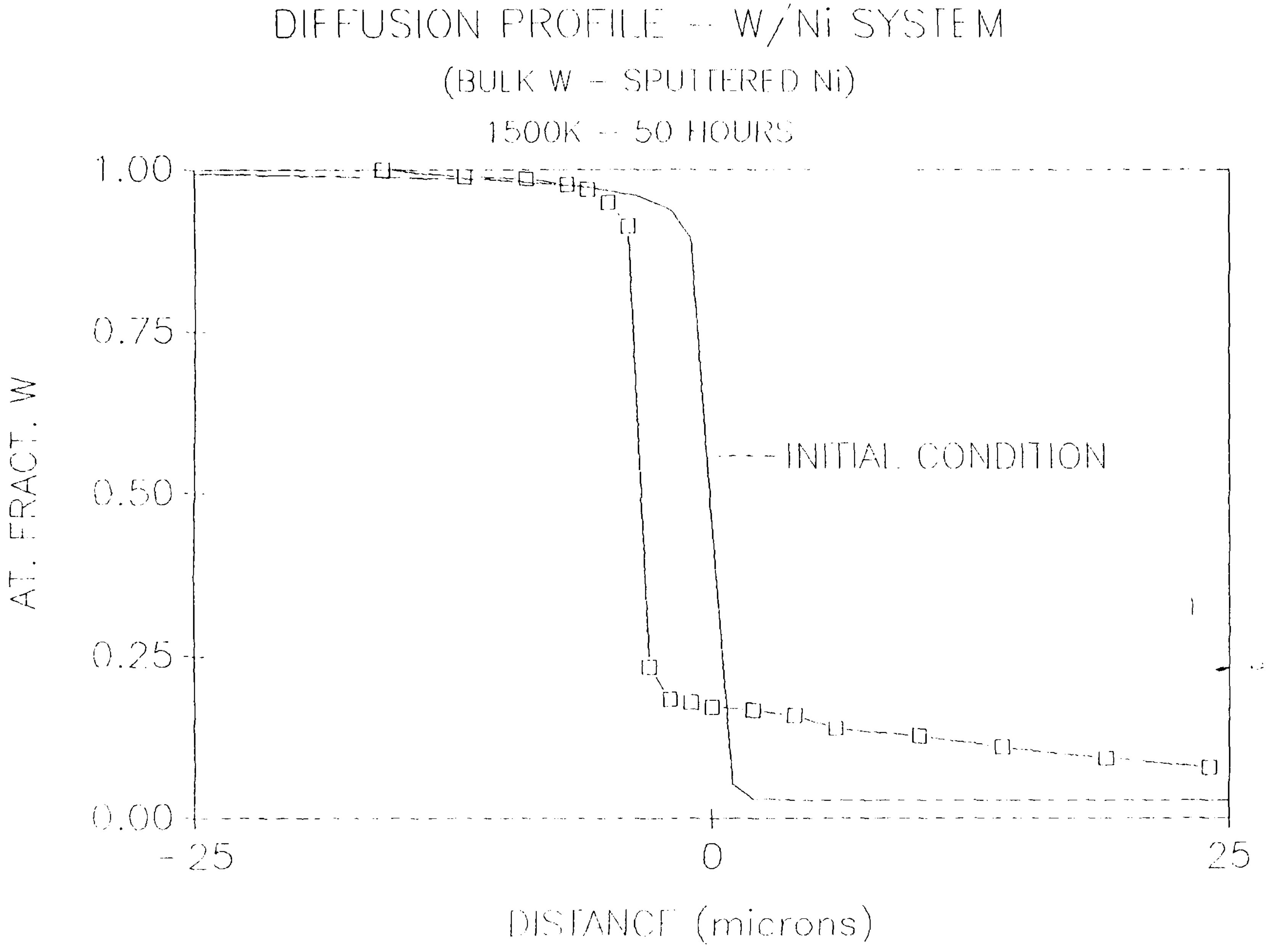

DIFFUSION PROFILE -- W/Ni SYSTEM
(BULK W - SPUTTERED Ni)
1500K -- 50 HOURS
INITIAL CONDITION
1.00
0.75
0.50
0.25
0.00
AT. FRACT. W
-25
0
25
DISTANCE (microns)

Figure 3

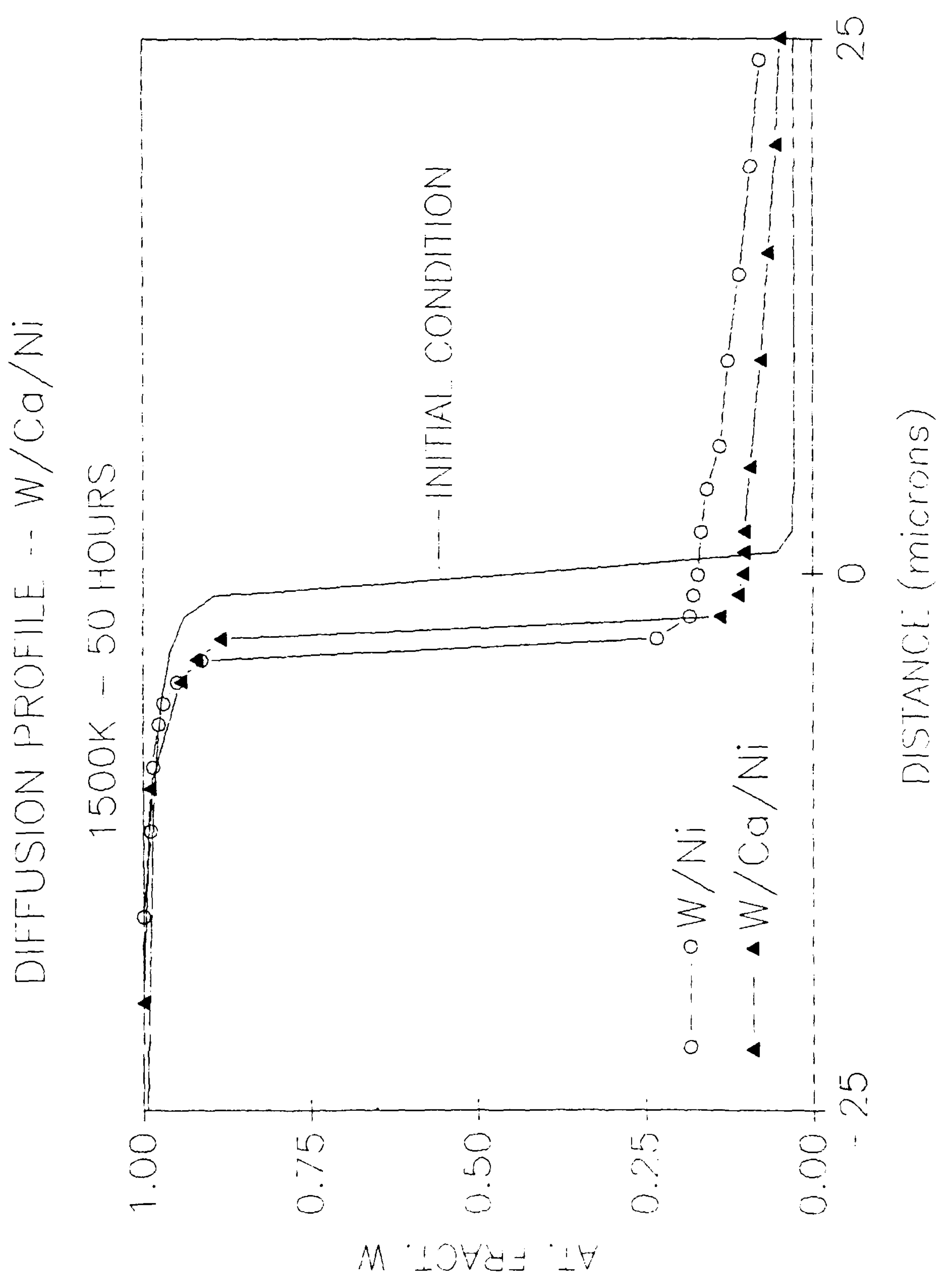
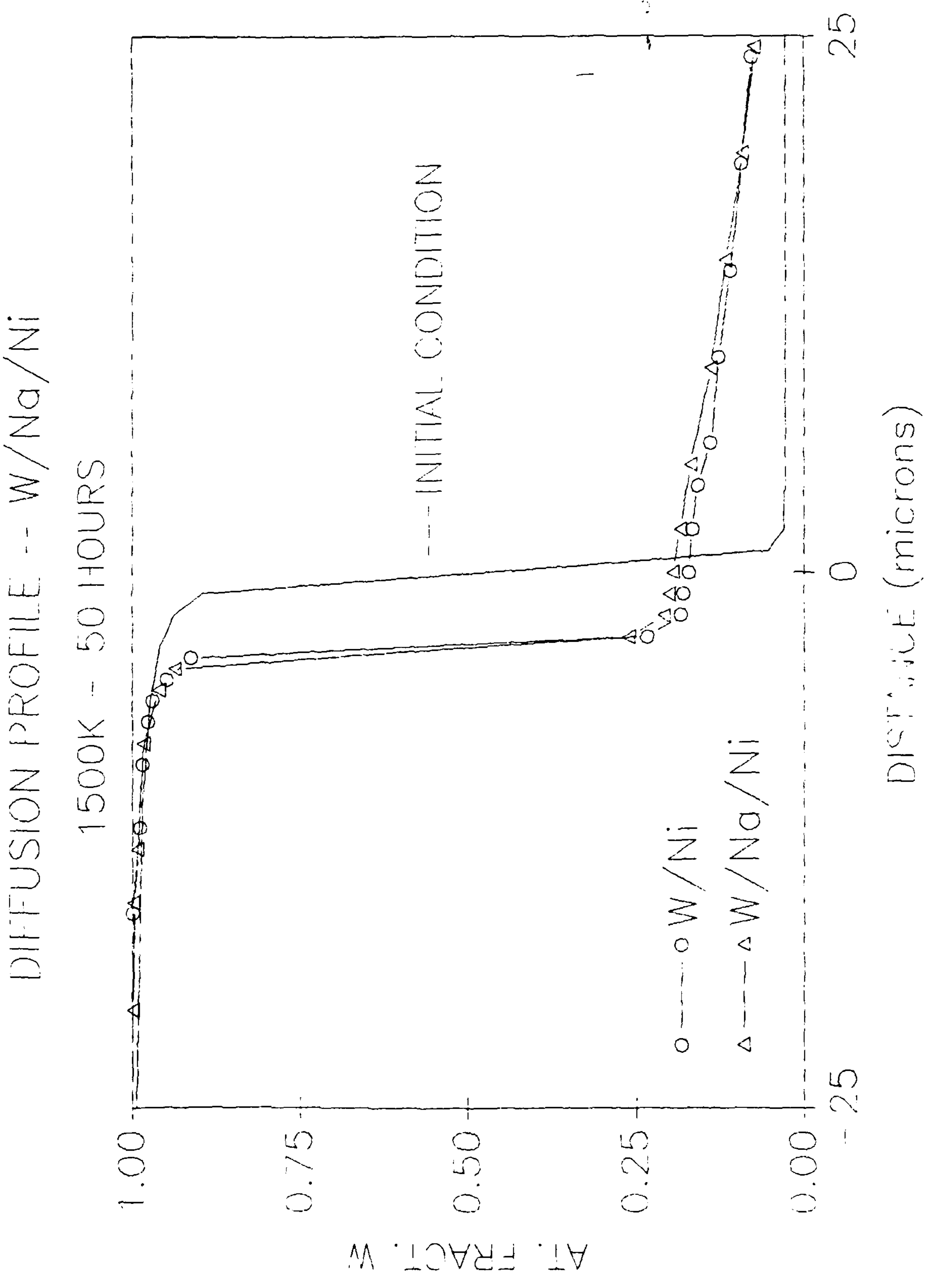

DIFFUSION PROFILE -- W/Ca/Ni
1500K - 50 HOURS
INITIAL CONDITION
W/Ni
W/Ca/Ni
AT. FRACT. W
1.00
0.75
0.50
0.25
0.00
-25
0
25
DISTANCE (microns)

Figure 4.


DIFFUSION PROFILE -- W/Na/Ni
1500K - 50 HOURS
INITIAL CONDITION
W/Ni
W/Na/Ni
1.00
0.75
0.50
0.25
0.00
AT. FRACT. W
-25
0
25
DISTANCE (microns)

accelerated diffusion for the limited conditions tested to date. This may be due to excessive local damage caused by the implantation of these large atoms in too thin a surface region.

The second method for application of alkali diffusion barriers being examined is a slurry technique utilizing an organic sol with an alkali suspension. Again, the W / Ni system is being used as our model. Incorporation of the barrier layers is accomplished by high speed spinning of the W substrate with gradual application of the barrier suspension, followed by a short anneal to allow the alkali atoms to diffuse into the tungsten surface and decompose the organic sol, and finally, the addition of the Ni layer. Attempts to deposit Ni by a electroless technique have had marginal results, therefore a industrial source has been contacted to fabricate the diffusion couples by hot-pressing. Once completed, these diffusion couples will be annealed along with an uncoated control couples in the temperature range of 1300 - 1500K so that the effectiveness of the barriers made by this technique may be assessed and compared to the ion implanted barriers.

Efforts in the area of diffusion barriers are continuing in both methods of fabrication. Although simple, it is clear that for a more quantitative evaluation of diffusion barrier effectiveness ion beam sputtered nickel layers will have to be replaced by material which has characteristics more closely resembling bulk material. We are pursuing sources who can diffusion bond all specimens for this aspect of the program, thereby ensuring reliable comparative data. To date, efforts in the slurry method have relied on sols saturated in salts of the alkali metals. Planned studies for the second year include variations in concentrations in the sol slurries as well as variations in the ion densities for the ion implantation process.

Our study of the interdiffusional stability and chemical compatibility of metal/intermetallic and intermetallic/intermetallic composite systems have focussed on the W/Ni_3Al and Ni_3Al/TiAl systems. These systems have been chosen for several reasons.

Although these systems show promise from an engineering viewpoint due to the availability and properties of commercial tungsten fibers and the expected oxidation resistance of aluminide matrix materials, perhaps more importantly, they enable a fundamental scientific analysis. By limiting the analysis to ternary systems (for both metal/intermetallic and intermetallic/intermetallic couples) the analysis of the interdiffusional kinetics and intermediate phase formation is greatly simplified. Also both Ni_3Al and TiAl are FCC based, being $L1_2$ and $L1_0$ respectively. As a consequence of these simplifications, results for metal/intermetallic and intermetallic/intermetallic diffusion may be compared and incorporated into a model to describe universal behavior in ternary systems. Several avenues of analysis are promising. For the simpler W/Ni_3Al system, a first effort of Boltzmann - Matano analysis may be easily employed by considering the system a pseudobinary and considering the diffusion path constant with temperature. Thus, the interdiffusional kinetics may be expressed in terms of W concentration and temperature in a simple fashion. The correlation of the resultant composition dependent interdiffusion coefficients with ordering parameters, as well as kinetics of formation of any intermediate phases (NiW,Ni_4W), should any form, should indicate what, if any, more complicated analyses might be called for. Possibilities include the cluster variation method (CVM), the path probability method (PPM), and Monte Carlo techniques.

We have encountered some problems with startup in this area of the program. Although Ni_3Al was rather easily acquired, TiAl has been difficult to obtained. An industrial source has agreed to melt a button for our use but this has delayed progress in our intermetallic/intermetallic efforts. There has also been some delay in fabricating the metal/intermetallic diffusion couples as we were required to find a second source to hot-press diffusion couples. These W/Ni_3Al couples should be completed shortly.

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