

MELTING BEHAVIOUR AND PHASE EQUILIBRIA IN THE SYSTEM NICKEL-CHROMIUM-TANTALUM

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ABSTRACT

Melting behaviour and phase equilibria of nickel rich Ni-Cr-Ta alloys have been investigated by means of differential thermal analysis (DTA) and electron-probe microanalysis (EPMA). As a result the projected areas of liquidus and solidus as well as the extent of the regions of primary, secondary and tertiary crystallization in the form of vertical sections (temperature-composition) could be determined. An isothermal section at 1000 °C as the result of the microprobe analysis describes the partition of the phase regions in the solid state adjoining to the solidus.

INTRODUCTION

Nickelbase alloys are first choice high-temperature materials because of the precipitation hardening ability and the good corrosion- and oxidation resistance. Interesting technological features may be adjusted by optimizing the alloy composition and the conditions of preparation. For systematic development of such multi-component-alloy-systems it is necessary to know exactly about the melting behaviour and the corresponding phase equilibria as a function of alloy composition and of temperature.

EXPERIMENTAL PROCEDURE

Differential thermal analysis (DTA) was carried out on a Netzsch DTA-apparatus 404 with SiC electric resistance furnace and Pt-Rh thermoelectric couples outside the samples. Specimens of Ni-Cr-Ta alloys (~1.5 g) were prepared by melting the pure materials (Ni, Ta 99.9 %; Cr 99.5 %) in an atmosphere of purified argon in the furnace of the DTA-apparatus using Al<sub>2</sub>O<sub>3</sub> crucibles.

After cooling below the solidus the DTA-measurements were realized at a heating- and cooling-rate of 5 K/min. Pure nickel (99.99 %) was used as active reference material (~1.5 g) in order to calibrate the thermoelectric couples.

The evaluation was carried out by means of extrapolated peak beginning and peak maximum of the recorded temperature difference vs. time curve and its derivation  $d/dt$ .

Phase compositions in the solid state were measured by electronprobe micro-

analysis (EPMA) on a Jeol JXA-50 A instrument. It was operated at 15 KeV analyzing energy- and wave length dispersive. Pure nickel, chromium and tantalum (99.99) was used as standard. The measured microprobe intensity data were corrected using a computer program magic IV /1/ modified according to Ruste /2/. The relative errors are estimated to be less than  $\pm 2\%$ . A minimum of 4 analyses of each phase was carried out in order to provide tie-line and tie-triangle data for the construction of isothermal sections (see Fig. 1). No evidence of concentration gradient within individual phases was found indicating a close approach to equilibrium.

## RESULTS AND DISCUSSION

The results of EPMA investigations are presented in the form of an isothermal section at 1000 °C (Fig. 1); mean values of the measured phase compositions are plotted in black dots. The investigated portion of the Ni-Cr-Ta phase diagram involves the following six phases:  $\gamma$ -nickel solid solution,  $\alpha$ -chromium solid solution,  $\text{Ni}_3\text{Ta}$  designated  $\gamma'$ ,  $\text{Ni}_2\text{Ta}$ ,  $\text{Cr}_2\text{Ta}$  and  $\text{NiCrTa}$  a ternary compound. These six phases enter equilibrium in the form of four 3-phase regions and eight 2-phase regions. Thereby one slim 2-phase region always separates two 3-phase regions.

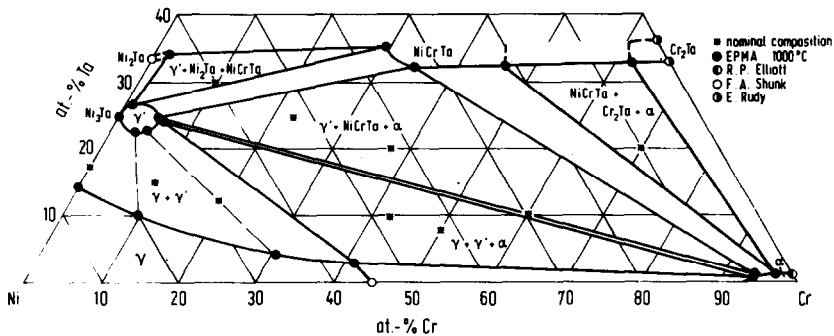


Fig. 1. Partial isothermal section of the system Ni-Cr-Ta at 1000 °C /3-6/

The nickel rich region of the system Ni-Cr-Ta is of technological interest for example with regard to precipitation hardening. In this region  $\gamma$ -solid solution is in equilibrium with  $\gamma'$ - $\text{Ni}_3\text{Ta}$  (precipitating phase) and  $\alpha$ -solid solution.

As the result of the DTA-investigations liquidus and solidus projections of nickel rich  $\text{NiCrTa}$  alloys are shown in Fig. 2. The liquidus surface (Fig. 2a) is characterized by the ternary eutectic point (10.5 at.-% Ta, 30 at.-% Cr:  $L \rightarrow \gamma + \gamma' + \alpha$ ) and by three binary eutectic reactions ( $L \rightarrow \gamma + \gamma'$ ,  $L \rightarrow \gamma + \alpha$ ,  $L \rightarrow \gamma' + \alpha$ ). These binary eutectic reactions are originated in the binary

systems Ni-Ta, Ni-Cr and in the 2-phase region of  $\gamma' + \alpha$ . In the ternary region they are visible as the intersection lines of the areas of primary crystallization of  $\gamma$ ,  $\gamma'$  and  $\alpha$ . Liquidus isotherms show the different bearing of the alloy constituents upon the liquidus temperature.

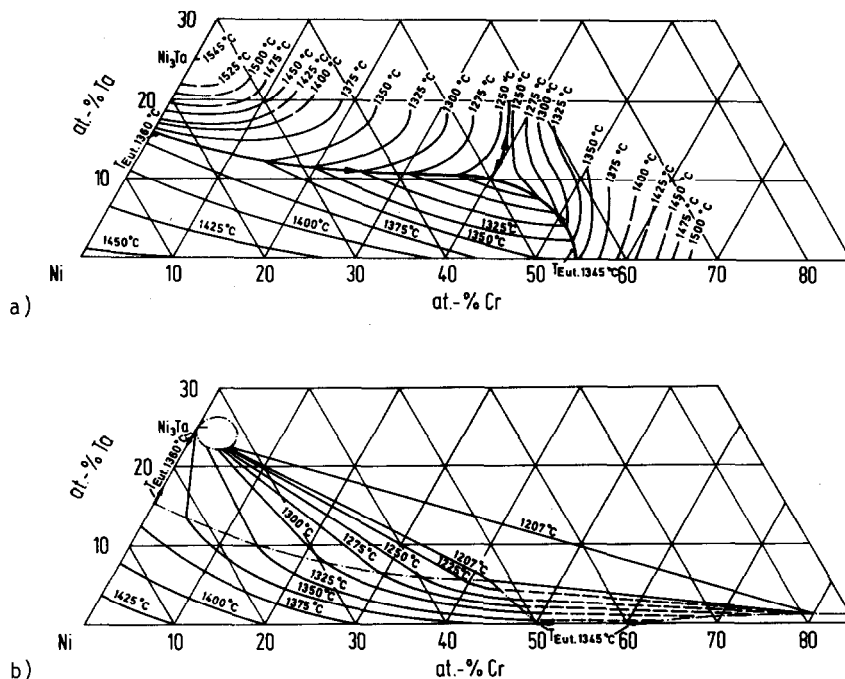


Fig. 2. a) Liquidus projection and b) solidus projection of nickel rich Ni-Cr-Ta alloys

The shape of solidus surface (Fig. 2b) has been constructed with regard to the corresponding phase equilibria in the solid state. Above the  $\gamma + \gamma' + \alpha$  3-phase region the ternary eutectic plane exists; above the 2-phase regions of  $\gamma + \gamma'$  and  $\gamma + \alpha$  the solidus surface is shaped helicoidal corresponding to the binary tie-lines. Dot-dash lines indicate the limitation of  $\gamma$ ,  $\gamma'$  and  $\alpha$  homogeneity regions.

Two vertical sections through the temperature-composition space diagram of the system Ni-Cr-Ta (Fig. 3) indicate the extend of the phase regions in the liquid and solid state as function of temperature measured by DTA and EPMA. Furthermore the regions of secondary crystallization ( $L + \gamma + \gamma'$ ,  $L + \gamma' + \alpha$ ,  $L + \gamma + \alpha$ ) as well as the planes of the nonvariant eutectic reactions ( $L \rightarrow \gamma + \gamma' + \alpha$ ,  $L \rightarrow \gamma' + \alpha + \text{NiCrTa}$ ) are visible.

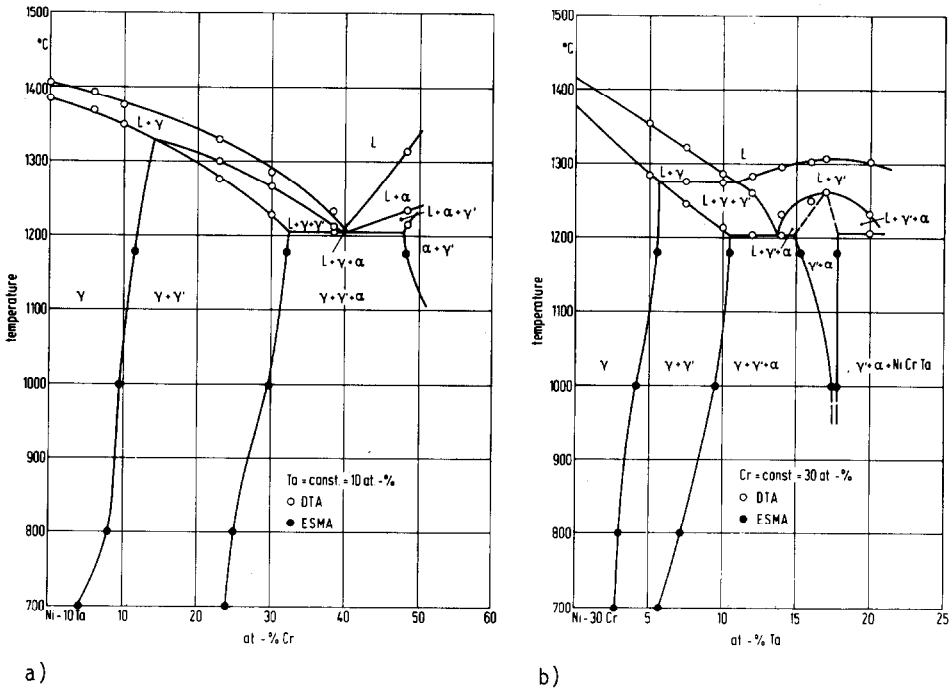


Fig. 3. Vertical section through the space diagram of the system Ni-Cr-Ta  
 a) at 10 at.-% Ta b) at 30 at.-% Cr

#### SUMMARY

Melting behaviour and phase equilibria of nickel rich Ni-Cr-Ta alloys have been investigated by means of DTA and EPMA. As a result the projected areas of liquidus and solidus and isothermal and vertical sections could be determined.

#### ACKNOWLEDGEMENTS

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#### REFERENCES

- 1 J.W. Colby, Adv. X-Ray Analysis 1968, 11, p. 287
- 2 J. Ruste, Microsc. Spectrosc. Electron, 1979, 4, p. 123
- 3 O. Knotek, S.U. Schittny, W.-G. Burchard, P. Karduck, Beitr. elektronenmikroskop. Direktabb. Oberfl. 1983, 16, p. 29
- 4 F.A. Shunk, Constitution of Binary Alloys, Mc Graw Hill, New York, 1969
- 5 R.P. Elliott, Constitution of Binary Alloys, Mc Graw Hill, New York, 1965
- 6 E. Rudy, Compendium of Phase Diagram Data, Part V, Air Force Materials Laboratory Report AFML-TR-65-2, 1969