Active and Passive Elec. Comp., 1989, Vol. 13, pp. 249–260
Reprints available directly from the publisher
Photocopying permitted by license only

Printed in Great Britain

A NEW STABILITY TEST FOR PASSIVATED NiCr THIN FILM RESISTORS

R. RIESENBERG, H. DINTNER

Akademie der Wissenschaften der DDR, Physikalisch-Technisches Institut, Helmholtzweg 4, Jena 6900, GDR

(Received December 2, 1987, in Final Form September 29, 1988)

A suitable short-time test for analysis of long term stability is presented for the case of passivated NiCr thin film resistors revealing an aging characteristic which is not of Arrhenius type. Based on the in-situ measurement of resistance change during a continuous temperature rise, so-called temperature ramp curve, a well-defined correlation is found between the film stability and a characteristic temperature T_p where the temperature ramp curve exhibits a maximum. In this way, a reliable prediction of the long term stability can be made within only a few hours. The influence of the heating rate on the characteristic temperature T_p is shown. Furthermore, it is experimentally proved that the values of T_p are not essentially determined by the reversible resistance changes due to differential temperature coefficient of resistance, but indeed by irreversible aging processes.

1. INTRODUCTION

The long term stability of resistors in one of the most important parameters for their application in microelectronics. Therefore, a reliable method is needed in order to characterize the stability within a short time and—if possible—to predict the long term stability under operating conditions. In the case of unpassivated NiCr thin film resistors an Arrhenius-like dependence of resistance change on temperature is found,[1,2] for that reason a simple extrapolation from high aging temperatures to lower ones provides the desired prediction. But, as shown in Fig. 1, NiCr thin films which have been passivated by a SiO_2 film and preaged in the usual manner to achieve a temperature coefficient of resistance (TCR) near zero, reveal a more complex aging behaviour. Especially the increase of temperature causes an inversion of the sign of resistance change. Consequently, a prediction of stability based on a straightforward extrapolation is not possible.

In this paper a stability test for passivated NiCr thin film resistors is presented which involves the measurement and analysis of resistance

change in dependence on a continuously rising temperature. This kind of thermal analysis is known in chemistry as thermoelectrometry[3] in order to investigate the properties of bulk materials. It has been already used in a similar way in order to determine the kinetic parameters of electromigration in thin metal films, so-called TRACE-technique.[4] Also, the step-annealing experiments made with various alloys to describe the establishment of atomic short-range order[5,6] can be interpreted as the measurement of the resistance change with (step-wise) variation of the temperature. But hitherto, there was no suitable method which gives a correlation between different temperature values in order to perform a prediction of the long term stability for films whose aging characteristic is not of Arrhenius-type.

2. EXPERIMENTAL

The NiCr films were reactively deposited by dc-magnetron sputtering in an oxygen containing argon atmosphere. Heated Si wafers covered by a thermally grown SiO_2 film of about 1.2 μm thickness served as substrates. The target compositions were Ni/Cr = 33/67 and 43/57 at%, respectively. The oxygen concentration within the films amounted to about 35 at% according to the adjusted ratio of the metal condensation rate and the oxygen partial pressure during deposition. Under these conditions an as-deposited resistivity of approximately 10^{-3} Ohmcm was obtained, i.e., the sheet resistance of the films with a thickness of about 40 nm was typically 250 Ohm/sq. After preparation of the resistor pattern by usual photolithography a passivation SiO_2 film of 1 μm thickness has been deposited by means of reactive dc-magnetron sputtering. For termination of the resistors contact holes were etched and a film sandwich with an Al conductor was deposited and patterned. In order to get a small TCR (which is technologically defined as the mean reversible resistance change per degree within the temperature region 300 . . . 400 K) the samples were exposed to a heat treatment for more than 25 hrs at a constant temperature of 630 and 700 K, respectively, corresponding to the two prepared film compositions. During this procedure the TCR changes from starting values of typically −150 and −100 ppm/K to about zero correlated with a monotonic decrease of resistance of −25 and −35 %, respectively.

In order to characterize the resulting aging behaviour of such films, further heat treatments like shown in Fig. 1 were additionally performed

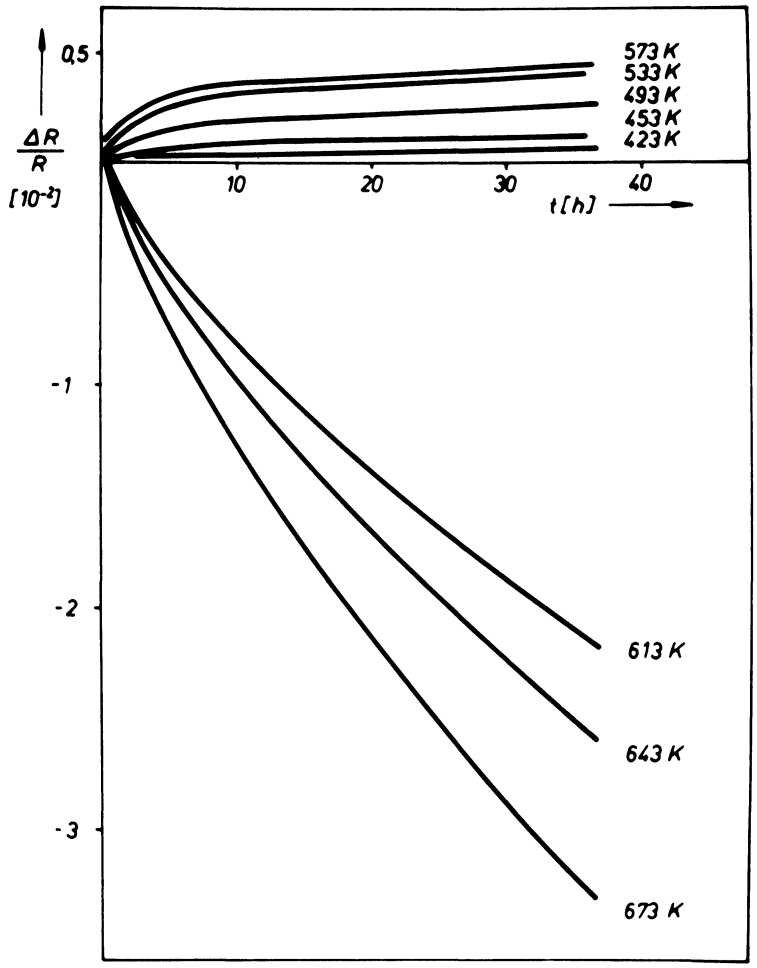

FIGURE 1 Resistance changes of passivated NiCr films versus time at different temperatures (isothermal aging); Ni/Cr = 43/57 at%.

at distinctively lower temperatures thus securing that the TCR remains nearly constant. Then the long term stability was determined for one part of the samples by measuring the resistance change under standard test conditions (400 K, 100 and 1000 hrs).

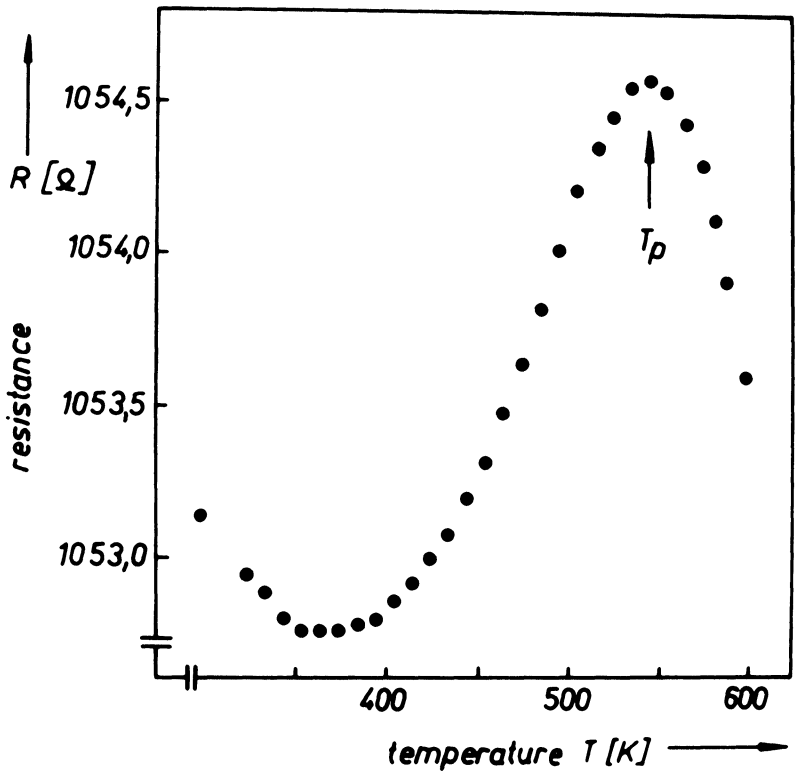

FIGURE 2 Resistance versus linear rising temperature (temperature ramp curve); Ni/Cr = 33/67 at%; heating rate $\beta = 1.7 \cdot 10^{-2}\ Ks^{-1}$ = const.

The other part of preaged thin film resistors were introduced into a special temperature chamber which allows an in-situ measurement of resistance by four probe technique at varying temperatures determined by a Pt resistance thermometer and controlled within ± 0.1 degrees. For the case of a linear temperature rise a typical curve of resistance change is shown in Fig. 2. The temperature ramp was $1.7 \cdot 10^{-2}\ Ks^{-1}$, therefore, for the temperature range 400 . . . 550 K which is usually used a measuring time of about 2.5 hrs is needed. It should be noted from Fig. 2 that for an exact determination of such ramp curves a resolution of resistance measurement of at least 10^{-5} is required.

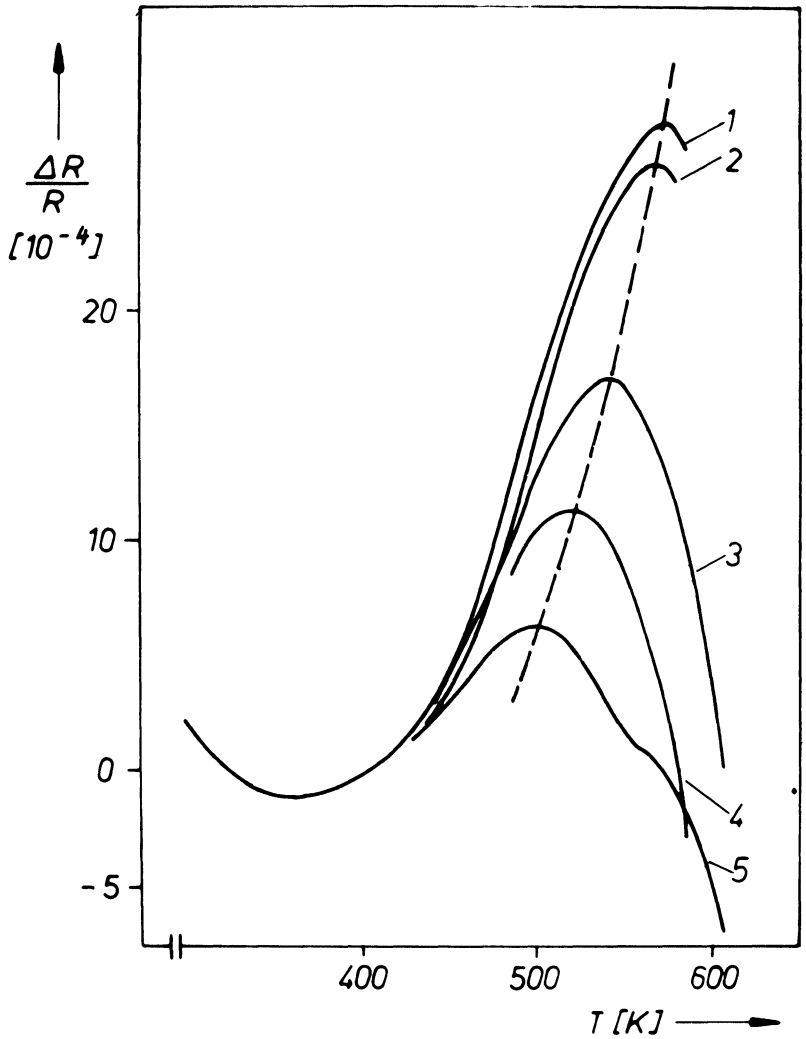

FIGURE 3 Temperature ramp curves of TCR adjusted films with Ni/Cr = 33/67 at% (related to T = 400 K) for (a) various additional isothermal treatments over 50 hrs at (1) 300 K, (2) 373 K, (3) 573 K, (4) 533 K, (5) 493 K; measured with $\beta = 1.7 \cdot 10^{-2}$ Ks^{-1} (b) different heating rates β (1) $1.7 \cdot 10^{-2}$ Ks^{-1}, (2) $8.5 \cdot 10^{-3}$ Ks^{-1}, (3) $4.25 \cdot 10^{-3}$ Ks^{-1} after constant additional isothermal treatment (493 K, 50 hrs).

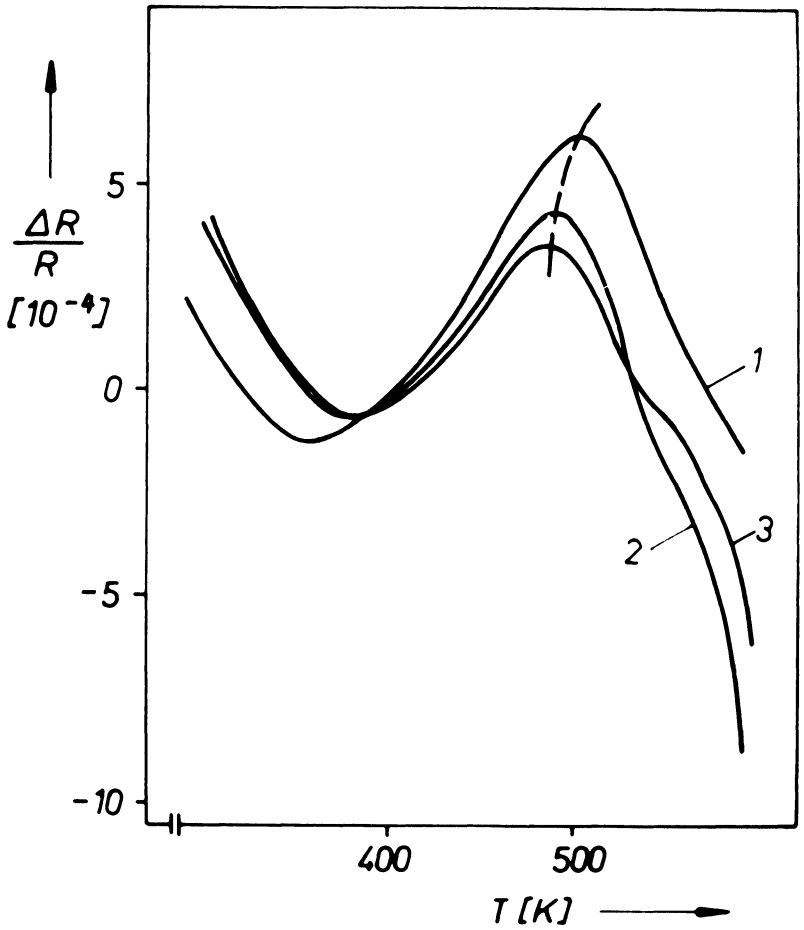

FIGURE 3b

3. RESULTS AND DISCUSSION

Figure 3 gives some characteristic features of temperature ramp curves $\Delta R/R = f(T)$, where $T = \beta t$, for various annealing treatments after TCR adjustment and for different heating rates β. At low temperatures up to about 420 K the curves essentially represent the reversible temperature dependence of resistivity with the nearly parabolic minimum which is

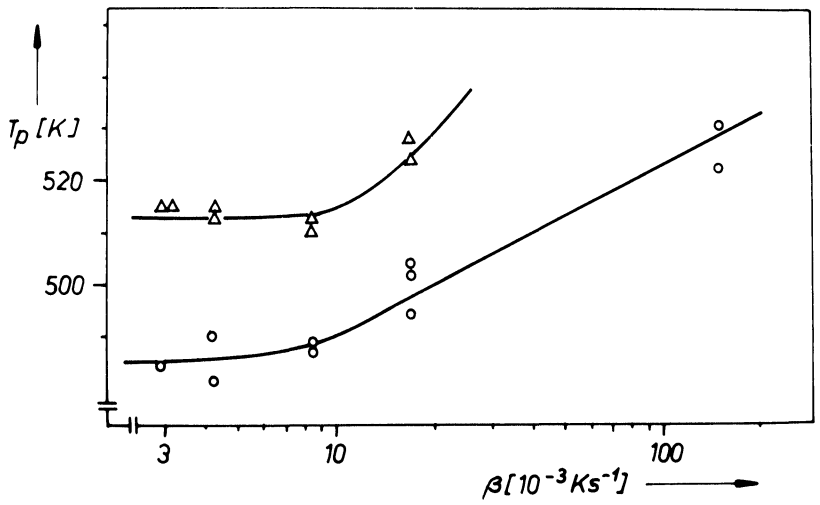

FIGURE 4 Dependence of characteristic temperature T_p on the heating rate β of two experimental runs (different additional isothermal treatments); Ni/Cr = 33/67 at%.

typical for NiCr thin film resistors and demonstrates the limited significance of the technological TCR. Above 420 K irreversible resistance changes due to aging processes are superposed and dominate more and more with rising temperature. Therefore, in this region the temperature ramp curves exhibit a marked hysteresis depending on the heating rate (see below).

The occurrence of a maximum at a characteristic (peak) temperature T_p which could be expected according to the sign inversion in Fig. 1 is of particular interest. The physical origin can be explained as follows: The temperature T_p directly reflects the steady state of the atomic short-range order established by the foregone heat treatments including the ramp experiment itself. As will be shown in detail in a paper under preparation, the resistance changes around the maximum are caused by the approach of the short-range order to the temperature-depending steady state with a varying kinetics. Consequently, the peak temperature T_p depends in a characteristic manner on both the procedure of pre-aging (Fig. 3a) and the heating rate β (Fig. 3b). But, if the heating rate is held constant or smaller than 10^{-2} Ks^{-1} (cf. Fig. 4), the T_p values can be attributed to the preparation conditions under consideration with the aim to use them as an indicator for the film stability.

It should be noted that the heights of the maxima in Fig. 3 are influenced

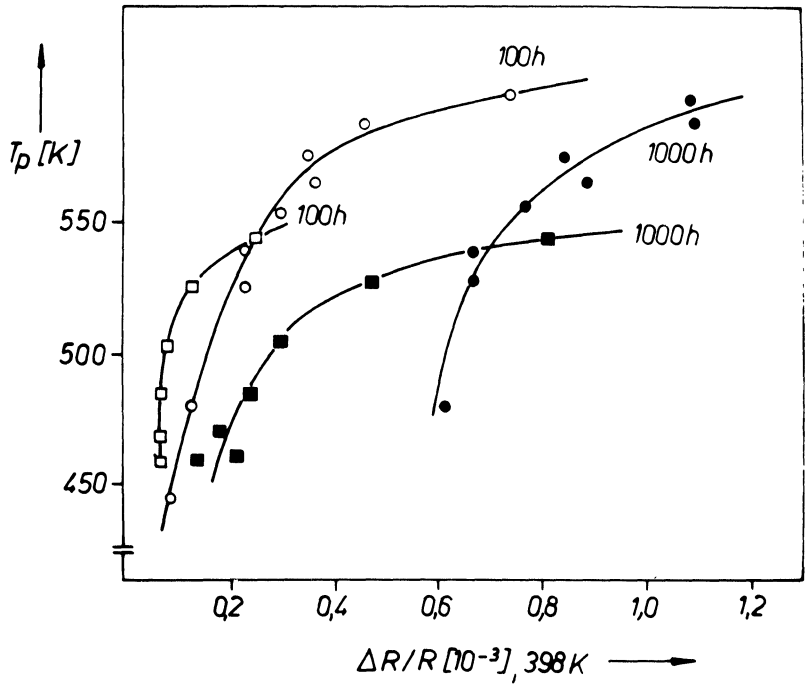

FIGURE 5 Correlation between characteristic temperature T_p and long term stability at 400 K after 100 hrs (empty symbols) and 1000 hrs (full symbols); Ni/Cr = 43/57 at% (circles) and 33/67 at% (squares); heating rate $\beta = 1.7 \cdot 10^{-2}$ Ks^{-1}.

by the technological conditions and cannot be analyzed with respect to the stability values in an unique manner, hitherto.

Furthermore, it has been proved that the peak temperature is nearly independent of the SiO_2 passivation film, i.e. for a given NiCr film the T_p values are only slightly changed (about 20 K) in spite of a variation of the SiO_2 thickness in the range of 0.05 . . . 1.5 μm or the kind of deposition technique (reactive DC and non-reactive RF sputtering).

Figure 5 demonstrates that indeed a rigid correlation between the long term stability of the resistors at 398 K and their characteristic temperature T_p exists for a constant film composition. Because of the limited resolution of the measuring equipment used, the correlation in Fig. 5 is experimentally verified only down to values of $T_p \simeq 450$ K: At lower temperatures, i.e. for film with higher stability, the temperature ramp curves could not be

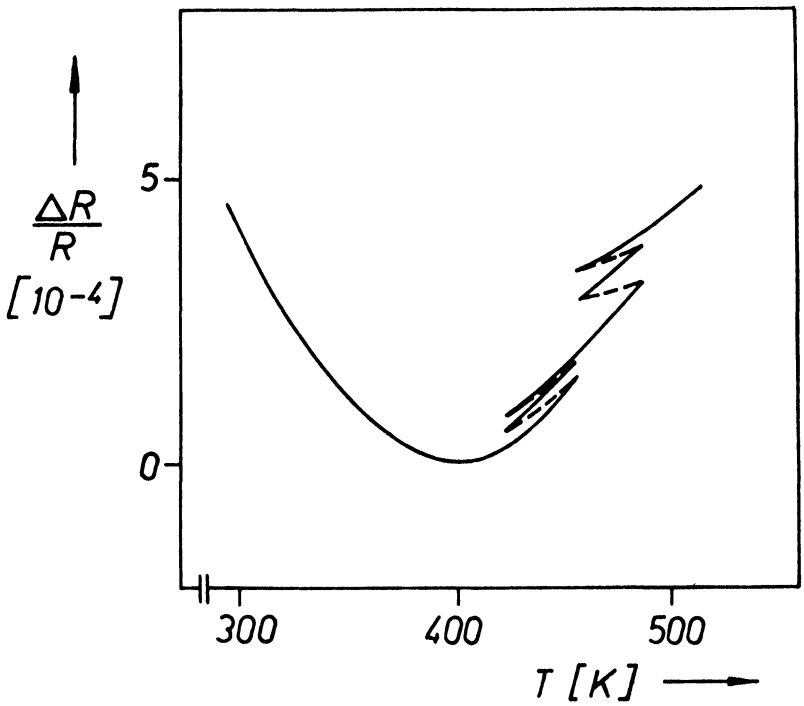

FIGURE 6 Separation of reversible and irreversible resistance change by periodical variation of temperature within $\Delta T = 35$ K (a) Principle of experiments: temperature ramp curve. (b) Slope of the periodically passed curve branch acc. to Fig. 6(a) at two temperatures: determination of reversible part (differential TCR) and irreversible one (α). (c) Irreversible resistance change per degree α versus temperature (same samples as in Fig. 6(b)).

measured with sufficient reliability even if the TCR of the film was restricted to values below 1 ppm/K. But nevertheless, in the examined range the determination of the characteristic temperature T_p gives a direct prediction of the long term stability.

Lastly, an important problem concerning the influence of reversible resistance change on the value of T_p has to be discussed. Above all, it must be proved that the characteristic temperature is mainly determined by aging processes and that eventual reversible parts due to the TCR can be neglected near T_p. As the NiCr films possesses in general a non-linear reversible temperature dependence, i.e. the differential TCR is not constant during the variation of temperature, a straightforward separation, like as-

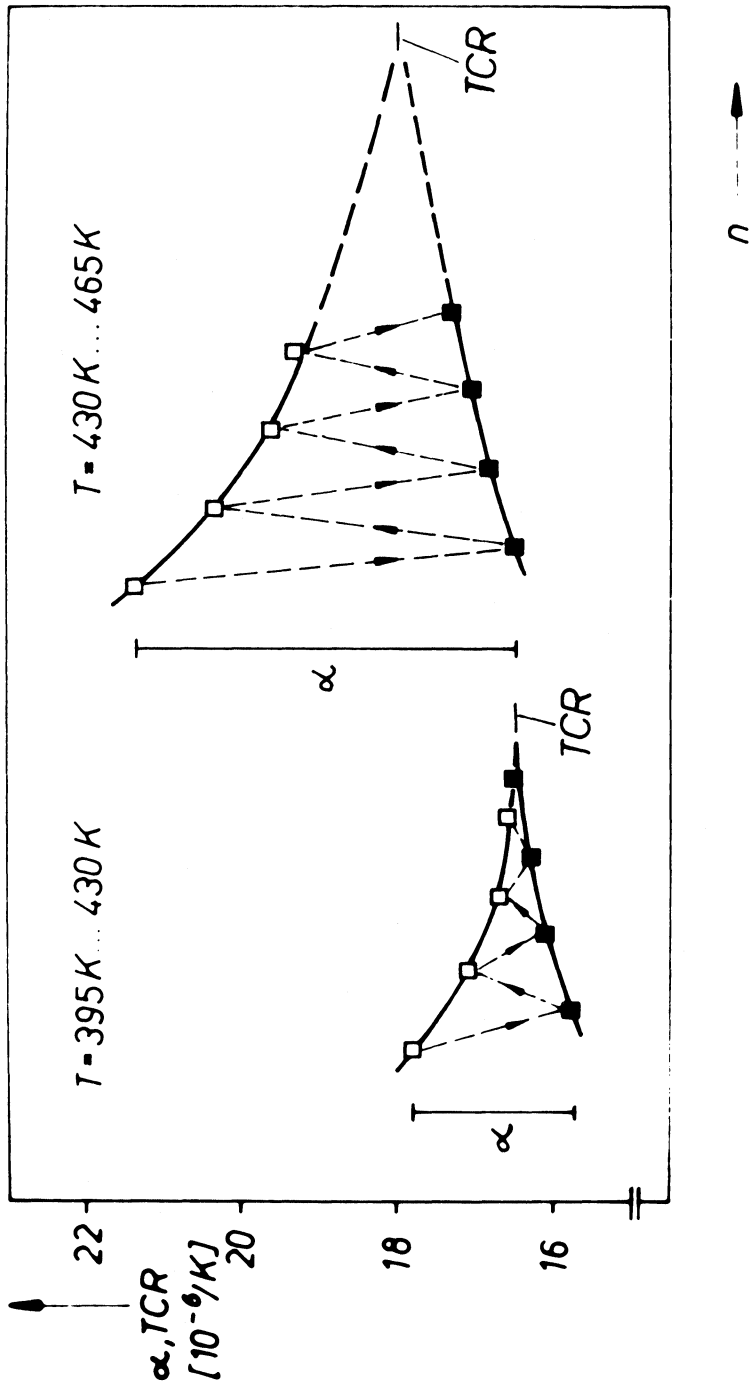

FIGURE 6b

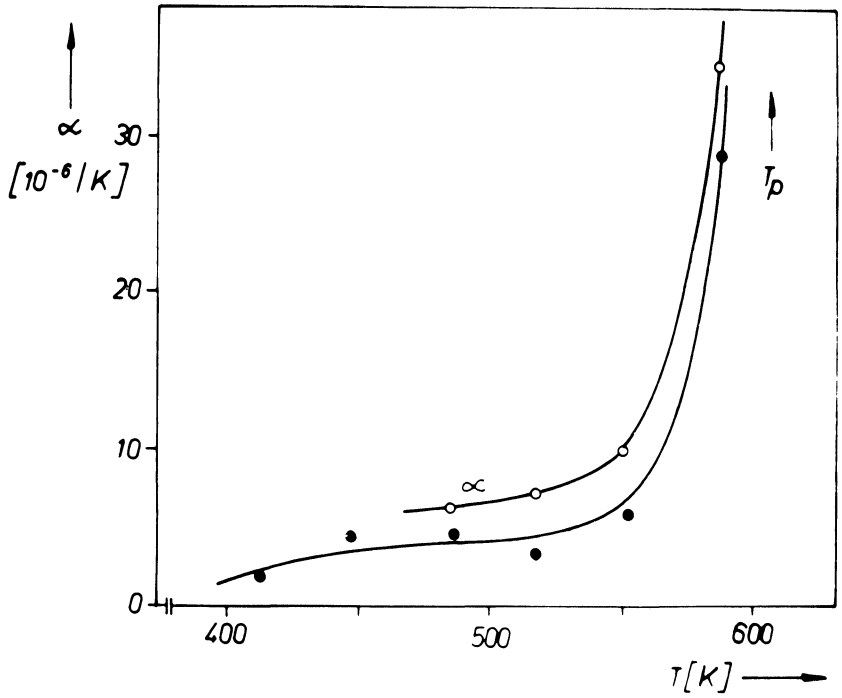

FIGURE 6c

sumed in case of Al films[4] is not possible. Therefore, some special experiments were performed by periodical variations of temperature within the small range of about 35 K during measuring the temperature ramp curve as schematically indicated in Fig. 6(a). The resulting changes of the slope of the ramp curve (resistance change per temperature, thus same dimension like TCR) are shown in Fig. 6(b) and provide the possibility to distinguish between the reversible part of slope due to TCR and the irreversible one denoted by α. The differential TCR remains nearly constant when the mean temperature of periodical variation is rised, i.e. far from the resistance minimum the NiCr films behave like a normal metal with an approximately linear temperature dependence. On the contrary, the corresponding irreversible part α strongly increases with rising temperature as shown in Fig. 6(c). Together with the fact that T_p does not depend on the heating rate β when $\beta \leq 10^{-2}$ Ks^{-1}, it can be concluded that the value

of the characteristic temperature T_p is essentially determined by aging processes and is, therefore, a suitable indicator for the film stability.

4. CONCLUSIONS

Based on the in-situ measurement of the resistance change during temperature rise, so-called temperature ramp curve, a well-defined correlation between the characteristic temperature T_p (where the ramp curve exhibits a maximum) and the long term stability is found for passivated NiCr thin film resistors. In this way, a new stability test could be established which allows a prediction within only a few hours and provides a suitable method to control and to improve the long term stability at a predetermined temperature.

Furthermore, it seems that the characteristic features of the temperature ramp curves which involve much more informations about the aging behaviour of the films than could be demonstrated by this paper, give a promising approach for a detailed study of aging processes in metal films.

ACKNOWLEDGMENT

The authors wish to thank Mr. K.-H. Wagner for the design of the temperature chamber for in-situ measurements and Dr H. Bartuch for preparation of samples.

REFERENCES

1. E. Salzmann, H. Delfs, Res. Rep. BMFT-FBT 76–26, July 1976, Munich, FRG.
2. H. Bartuch et al. "Aging Model of Reactively Deposited Thin Ni-Cr Films" Thin Solid Films *116*, 211 (1984).
3. C.B. Murphy "Thermal Analysis" Analyt. Chem. *50*, 143 (1978).
4. J.A. Schwarz "Determining Electromigration Kinetics of Thin Film Interconnects" Semicond. Internat., March 1985, p. 96.
5. R. Reihnser, W. Pfeiler "Atomic Short-Range Order in CuMn Alloys" J. Chem. Solids *46*, 431 (1985).
6. W. Pfeiler, R. Reihnser "On the Importance of Thermal pre-Treatment for the Microstructural Interpretation of CuMn Alloys" J. Phys. F: Met. Phys. *15*, 2547 (1985).