High Temperature Thermomechanical Properties of Titanium-Rhodium-based Alloys Containing Scandium

Unusual shape memory effects observed in scandium-substituted alloy system

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At high temperatures, the equiatomic binary compounds formed by Groups 4 and 8 transition metals are known to undergo martensitic transformation, which may be accompanied by a shape memory effect. Among these compounds, titanium-rhodium (TiRh) is of special interest not only because it undergoes two martensitic transformations at high temperature, for one of which the shape memory effect has been observed, but also because it demonstrates unusual shape recovery behaviour at temperatures higher than 400ºC. The present work focuses upon the thermomechanical and mechanical properties of 50 at% rhodium-scandium-titanium ternary alloys where Ti is substituted by Sc. These alloys were investigated for the first time using electrical resistance, dilatometry and three-point bending techniques in the temperature range 20ºC to 850ºC. It was found that the sample with 0.1 at% Sc exhibited full shape restoration in the ranges of both martensitic transformations at ~340ºC and ~750ºC. Two-way shape recovery was also observed. A small temperature hysteresis, desirable for alloys used in actuator applications, is present in TiRh and Rh-Sc-Ti alloys. Both TiRh and Sc-containing alloys exhibit continuity of the deformation process on cooling and shape restoration on heating in a wide range of temperatures. This feature of both TiRh and Rh-Sc-Ti alloys implies the possibility of their application in different heat-regulating elements at temperature ranges from room temperature to 850ºC.

Introduction

An important field for the application of commercial alloys with shape memory effect is heat regulation. The alloys considered in this paper are being investigated for use in this application at high temperatures. Thermomechanical regulators have a number of advantages over electromechanical and electronic regulators, which are prone to failure under certain conditions. Regulators made from shape memory alloy, such as locking devices for control rods in

nuclear power stations, could provide more robust safety systems as they do not require any external power source for their activation, for example.

One of the challenges in high-temperature materials science is to discover alloys that exhibit a high-temperature shape memory effect and to investigate the conditions, including temperature range, under which these properties are observed. The Rh-Ti system is considered promising taking into account information on the alloys based on equiatomic phase (1–5) and data on the thermoelastic properties of the equiatomic TiRh alloy at high temperatures (6).

There is limited literature data on the character and temperatures of transformation in near-equiatomic Rh-Ti binary alloys. Such data are to a certain extent conflicting, and concern mainly the crystal structure of the phases formed (1–5). An X-ray study of TiRh in the temperature range from room temperature to 1000ºC showed that a high-temperature phase with a caesium chloride (CsCl) type cubic crystal structure transformed to a phase with tetragonal structure at 845 ± 20ºC when cooled from 1000ºC, then transformed to a monoclinic phase at 83 ± 5ºC (3). Both transitions were considered as second order transitions. It was noted that no domain of coexistence of cubic and tetragonal phases was observed, while the tetragonal character of the phase structure increased on cooling gradually from 845ºC to 83ºC. At temperatures lower than 83ºC further distortion of the crystal lattice toward monoclinic symmetry took place.

Observed temperature intervals of three modifications of TiRh (3) confirmed previously published data (2) that the TiRh phase crystal structure at 700ºC is tetragonal and of the AuCu type, while at room temperature the phase is monoclinic with lattice parameters different from those previously given (3). In alloys with compositions deviating from stoichiometry towards 10 at% Rh content, according to the Rh-Ti phase diagram, a phase with orthorhombic crystal structure was observed at room temperature (2) within the homogeneity range of the equiatomic phase (4, 5).

Two transformations in TiRh were revealed by an electrical resistance method with temperatures that differ from those previously obtained (3, 6) (**Figure 1(a)**). Both transformations occurred almost without hysteresis. The fact that they are clearly separated by temperature (6) puts in doubt the interpretation of the transformation as a second order transformation (3).

The shape memory effect accompanies the formation of a phase with a monoclinic crystal structure in TiRh at temperatures close to those obtained by the electrical resistance method, M_s^2 ~340ºC. Anomalous behaviour of the sample when heated to 400ºC has been observed (6).

The aim of the present paper is to study the influence of the third component, Sc, on the properties of TiRh-based alloys and particularly the thermomechanical behaviour of these alloys in the temperature range from ambient to 850ºC. Sc was chosen to substitute Ti because it is similar to Ti by chemical properties and also forms an equiatomic compound with Rh. However the crystal structure of ScRh, which is of the CsCl type (the same as the TiRh parent phase), is stable from subsolidus temperature to room temperature (7). This makes it easy to follow changes that occur in the Rh-Sc-Ti ternary alloys with increasing scandium content.

Information on the effect of scandium on the martensitic transformation (MT) in TiRh is almost unknown. It was noted earlier that substitution of Ti by Sc would lead to a decrease of the martensitic transformation temperature (8). The phase relations in the ScRh-TiRh system have been studied elsewhere (9).

Experimental

The starting metals used for alloying were iodised Ti, Rh powder (99.97 wt%), distilled Sc to prepare the alloys with 0.1 at% and 1 at% Sc, and Sc metallic powder (chemical analysis: 1.3 wt% O and less than 0.15 wt% of metal admixture) to prepare the remaining alloys. Before melting the alloys, the rhodium powder was sintered in vacuum at 1200ºC and melted in an arc furnace in order to avoid sputtering during alloying. The alloys with 0.1 at% and 1 at% Sc were melted directly from the components and those with 2.2 at%, 3.5 at%, 16.7 at% and 21 at% Sc were melted from the ligature (the Sc-Rh binary alloys) with additions of titanium in an arc furnace with a non-consumable tungsten electrode on a water-cooled copper hearth under an atmosphere of purified argon gas. The ingots were melted four times to ensure a complete melt. The weight losses on melting were small (below 0.5 wt%) so nominal compositions are reported.

Samples of the six alloys were prepared along the ScRh-TiRh section. The as-cast alloys were investigated by electrical resistance and dilatometry tests. The electrical resistance was measured by the four-probe method with continuous heating of the sample at a rate of 20ºC min^{-1}. The change in sample length ($l_{20ºC} = 14.7$ mm) on heating and cooling in the dilatometry test was measured by an induction micrometer 'Micron-02'
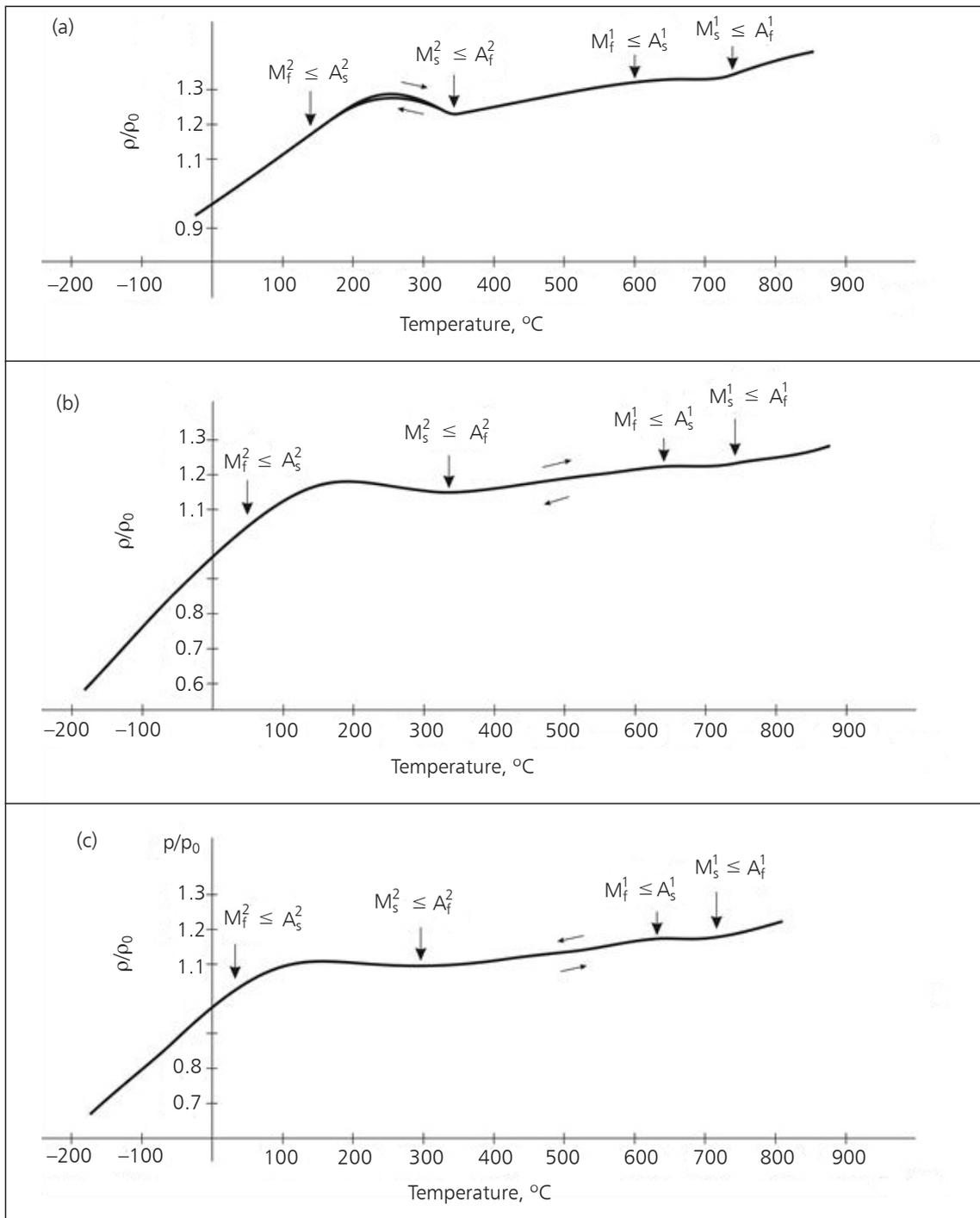
Fig. 1. Dependence of electrical resistance on temperature in ScRh-TiRh alloys: (a) TiRh; (b) Rh-0.1 at% Sc-Ti; and (c) Rh-1 at% Sc-Ti

supplied by All-Union company 'Stankoimport', Moscow, Russia, with accuracy ±0.1 micron. The temperature of each sample was measured with a chromel-alumel thermocouple welded to the sample that was placed in a quartz holder. Thermomechanical properties of the alloys were examined using the three point bending technique by thermocycling through the transformation range during cooling under load and

heating after unloading (10). Thermomechanical curves were taken several times for each sample at different loads. The sample sizes were $0.4 \times 0.4 \times 10$ mm (alloys with 0.1 at% and 1 at% Sc) and $0.4 \times 1.5 \times 8$ mm (alloys with 2.2 at% and 3.5 at% Sc). The loads varied between 50 g and 375 g. The sample of the alloy with 0.1 at% Sc underwent more detailed investigation in the temperature range which covered both transformations according to data on electrical resistance. The sizes of the sample and the load applied in this experiment are presented and discussed together with the results. The alloys were characterised by X-ray diffraction (XRD) and microstructure analyses and the results are presented elsewhere (9).

Results and Discussion
Electrical Resistance

The TiRh resistance curve shows two transformations in the solid (**Figure 1(a)**). The transformation at ~750ºC (M_s^1), of small intensity and without hysteresis, is indicated by a point of inflection on the curve of resistance. The transformation at ~340ºC (M_s^2), of greater intensity, occured with a small hysteresis: the change in electrical resistance reached ~10%.

Investigation of MT in the ScRh–TiRh ternary alloys by the electrical resistance method was carried out on the samples with 0.1 at%, 1 at%, 2.2 at%, 3.5 at%, 16.7 at% and 21 at% Sc (**Figures 1(b)**, **1(c)** and **2(a)–2(d)**). The resistance curves for the first four alloys exhibit two transformations as was observed in the TiRh alloy. The heating and cooling curves of the alloys with 0.1 at% and 1 at% Sc almost superimpose in a wide temperature range (**Figures 1(b)** and **1(c)**). The effect that corresponds to the second transformation is more pronounced in these alloys, although unlike that in TiRh the transformation became almost without hysteresis. Given that the temperature of both transformations slowly decreased with increasing content of scandium in the alloys, the resistance curves were observed to behave similarly to TiRh.

In contrast to the curves of TiRh and the alloys with 0.1 at% and 1 at% Sc, a larger change in resistance with decreasing temperature was observed in the curves for the alloys with 2.2 at% and 3.5 at% Sc (**Figure 2**). The first martensitic transformation (M_s^1) for these alloys was clearer and more intense (**Figures 2(a)** and **2(b)**). Its temperature varied little with increasing scandium content. At the initial transformation stage (740ºC–705ºC) a sharp change in electrical resistance took place which is a characteristic of the transformation and gives reason to believe that in the

process of structural transformation an intermediate phase with a crystal structure similar to the R-phase in TiNi might form first (premartensitic transformation) (11). The second MT was identified by a change in the slope of the resistance curve. The critical temperatures of the martensitic points in the forward and reverse transformations coincide.

With increasing scandium content in the alloys to 16.7 at% Sc only one transformation was observed in the resistance curve at approximately 400ºC, a temperature significantly lower than the first transformation in alloys with lower scandium content (**Figure 2(c)**). The shape of the alloy curve on cooling repeated that for alloys with 2.2 at% and 3.5 at% Sc (**Figures 2(a)** and **2(b)**). Tests on the alloy with 16.7 at% Sc at temperatures below room temperature (down to −196ºC) showed the absence of any effect. This confirms the assumption that with increasing content of scandium in the alloys the second transformation disappeared instead of lowering its temperature. According to X-ray analysis this alloy has a tetragonal crystal structure of AuCu type at room temperature (9). Thus, it may be concluded that the effect observed on the resistance curve of the alloy with 16.7 at% Sc corresponds to the structural transformation CsCl → AuCu. Given the above as well as data on the electrical resistance of TiRh and the alloys with lower scandium content, even without direct studies of the structure by high-temperature X-ray analysis, it seems clear that the product of the first MT in all alloys of the ScRh-TiRh system is a phase with a tetragonal crystal structure of the AuCu type.

Only a little variation in the slope of the resistance curve of the alloy with 21 at% Sc was observed at about 200ºC, indicating that the intensity of the transformation decreases with increasing content of scandium in the alloys (**Figure 2(d)**). This alloy is two-phased at room temperature: CsCl + AuCu (9).

Thus, according to electrical resistance studies, substitution of Sc for Ti in TiRh resulted in inhibition of the second MT, the product of which would be a phase with monoclinic crystal structure, and in lowering the characteristic points of the first transformation, which disappeared with increasing content of scandium in the alloys.

The electrical resistance data concerning the two stage transformations in the solid Rh-Sc-Ti ternary alloys and similar data on TiRh point to the existence of a two phase region in these alloys and are at variance with earlier conclusions (3) on the attribution of the transformation in TiRh to second order transformation.
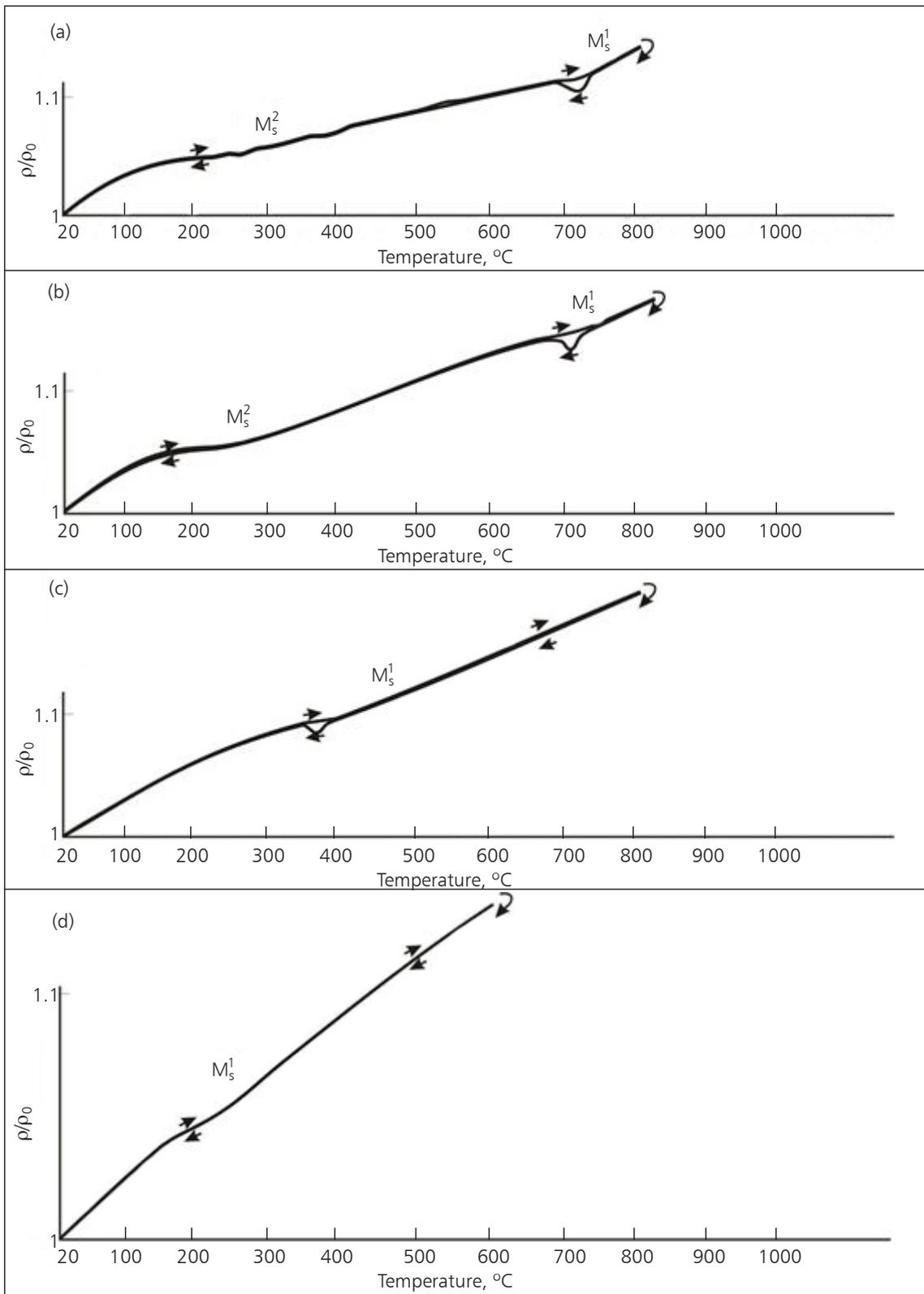
Fig. 2. Dependence of electrical resistance on temperature in ScRh-TiRh alloys: (a) 2.2 at% Sc; (b) 3.5 at% Sc; (c) 16.7 at% Sc; and (d) 21 at% Sc

X-ray analysis of the ScRh-TiRh alloys with 0.1 at%–16.7 at% Sc showed regions of coexistence of phases of TiNi and AuCu types as well as those of AuCu and CsCl types (9). The electrical resistance data on TiRh-TiCo alloys in which rhodium atoms were replaced with cobalt atoms also demonstrated two stages of transformation that were distinctly separated (6). In addition, some effects related to both transformations have been observed on the thermal analysis curve of TiRh (9). All these data strongly suggest that the transformations in TiRh and alloys based on it are first order.

Thermomechanical Tests

Thermomechanical tests were carried out for the alloys with 0.1 at%, 1 at%, 2.2 at% and 3.5 at% Sc (**Figures 3–5**). There was a continuous bend for the sample with 0.1 at% Sc cooled under a load of 50 g from ~900ºC–50ºC, and this was more intense in the range 750ºC–650ºC. On heating (after unloading) the sample recovered its shape completely. In the range 400ºC–200ºC the degree of deformation on cooling increased almost linearly. At a load of 100 g a bend occurred in the same temperature range (**Figure 3(a)**, dashed lines) and the deformation of the sample proved to be twice as large in comparison with that at a load of 50 g; however the sample recovered fully on subsequent heating. Some residual plastic deformation that occurred after loading and unloading of the sample at ~850ºC indicated the inconsistency of the applied load with the elastic parameters of the sample at this temperature. Note the basic similarity of the thermomechanical curves of the alloy at different loads (**Figure 3(a)**).

The first transformation in the alloy containing 1 at% Sc occurred in the same temperature range, 750ºC–650ºC (**Figure 3(b)**). In the temperature range from 650ºC to room temperature the thermomechanical curve was almost linear. After unloading at 20ºC and subsequent
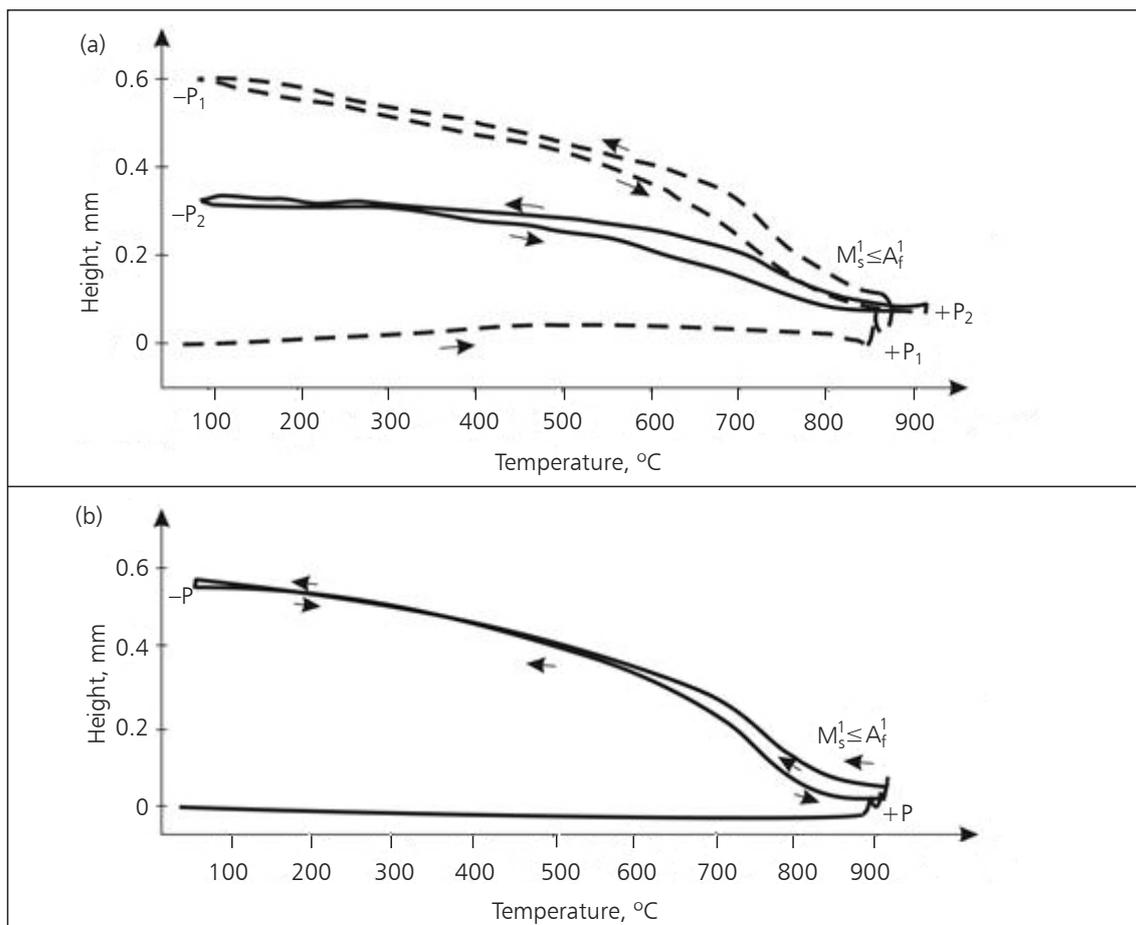

Fig. 3. Dependence of bending on temperature in ScRh–TiRh alloys: (a) 0.1 at% Sc; and (b) 1 at% Sc
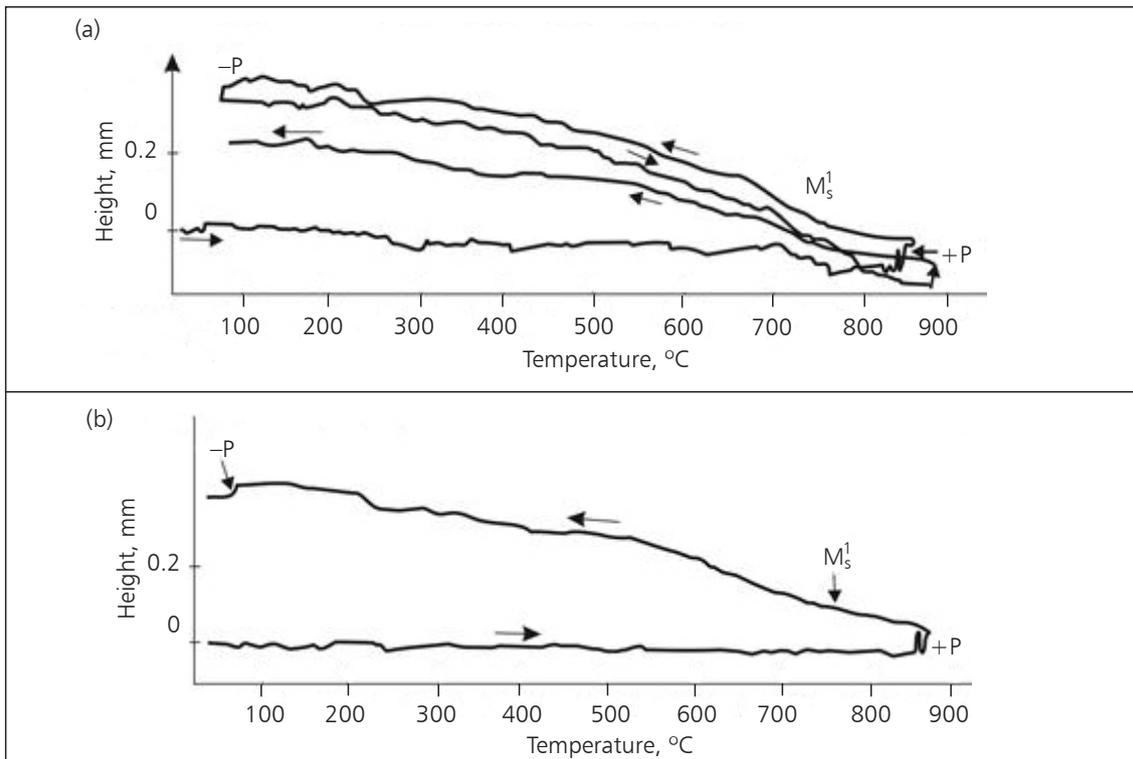
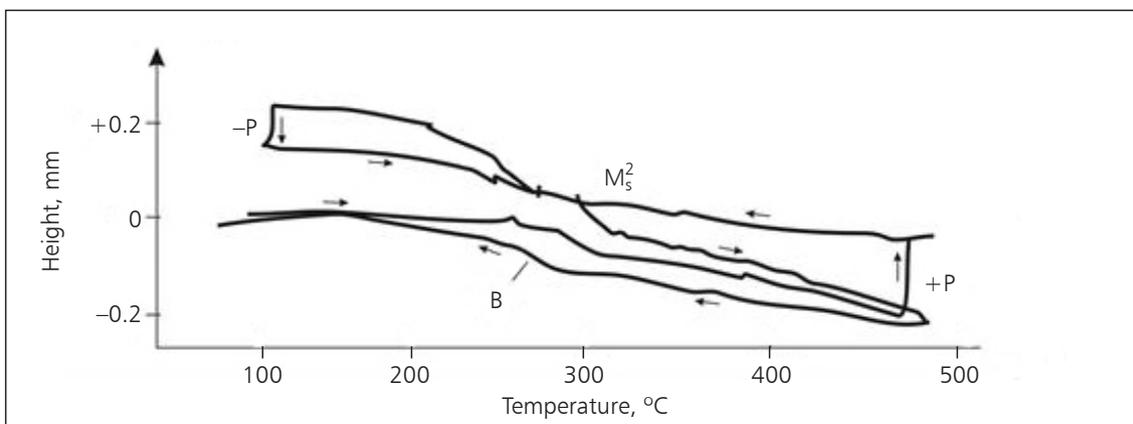Fig. 4. *Dependence of bending on temperature in ScRh–TiRh alloys: (a) 2.2 at% Sc; and (b) 3.5 at% Sc*

Fig. 5. *Dependence of bending on temperature in TiRh alloy*

heating to 850ºC, the sample's shape restored fully. Cooling under load to the temperature of liquid nitrogen (–196ºC) showed that the M_f temperature of the alloy was about 20ºC. Below this temperature cooling did not lead to additional deflection of the sample. The thermomechanical curves of both alloys were without hysteresis except in the MT temperature range for the transition CsCl → AuCu, where a small hysteresis occurred (**Figures 3(a)** and **3(b)**).

The thermomechanical curves of the alloys with 2.2 at% and 3.5 at% Sc are similar to those of the alloys with 0.1 at% and 1 at% Sc (**Figures 4(a)** and **4(b)**). The curves in **Figures 3–5** show the results of the thermomechanical experiments. They show the presence or absence of the shape memory effect and the unusual feature of the expansion of shape recovery at temperatures between 20ºC–700ºC. Such a feature has not been observed in other alloys exhibiting a shape memory effect.

The unusual performance of TiRh alloy observed during a study of its thermomechanical properties has already been mentioned (6). No further experiments had been carried out to study the sample's behaviour when heated above the temperature of 400ºC and it was only noticed that on reducing the elastic constants of the alloy with temperature, the bend of the sample would remain constant or increase with increasing temperature. Instead, during experiments further straightening of the sample was observed.

In order to study this anomalous behaviour, further tests were carried out during the present study on a sample of the ternary alloy with 0.1 at% Sc composition, chosen for being closest to TiRh. The similarity of the resistance curves of the two alloys (**Figures 1(a)** and **1(b)**) together with the proximity of their compositions is believed to justify treating the test results obtained for the 0.1 at% Sc alloy as being comparable to those expected for the TiRh binary alloy. **Figure 6** presents thermomechanical test curves of the 50 at% Rh–0.1 at% Sc-Ti alloy in different temperature ranges. The full bending curve taken in the range 20ºC–900ºC for the sample with parameters $0.5 \times 1.3 \times 8$ mm repeated the curves that were obtained in previous experiments (**Figures 3(a)** and **6(a)**): under constant load ($P_1 = 250$ g) in the temperature range 750ºC–20ºC continuous bend on cooling and subsequent full shape restoration on heating of the sample were observed. More intense bending took place in the temperature range of the first MT compared to that in the range of the second MT. While approaching the M_s^1 point on the cooling curve, a distinct bend was observed. This might be caused by the appearance of deformation martensite overtaking the tetragonal thermal martensite at the temperature M_s^1 (**Figure 6(a)**, A).

The fact that the contribution of the second MT to the overall bending is smaller than the first might be due to insufficient loading. With decreasing temperature, the coefficients of elastic deformation of all metals and alloys increase. Therefore for more accurate detection of bending in the temperature range of the second MT a bigger load of 600 g was used for the same sample. A partial bending curve for the interval of the second transformation is shown in **Figure 6(b)**. A partial bending curve for the first transformation for another sample with parameters $0.9 \times 1.4 \times 8$ mm and a load of 250 g is shown in **Figure 6(c)**. Applying a load greater than 250 g was not possible, due to expected plastic deformation at 850ºC when the alloy is in a high temperature phase with a cubic crystal structure.

These partial curves demonstrate full shape restoration and almost no hysteresis between the forward and reverse transformations. Alloys exhibiting transformation with a very small temperature hysteresis are known to be very useful for actuator applications (12). Another quality of thermomechanical behaviour of the alloy with 0.1 at% Sc was observed earlier for the TiRh binary alloy in the range of the second MT, which at the time was not explained (6). Upon heating of the sample free of load gradual spontaneous bending took place, followed by outward bending on subsequent cooling throughout the temperature range corresponding to the second transformation without a load (**Figure 5**, B). A bend on cooling and heating curves of the unloaded sample of the ternary alloy with 0.1 at% Sc was also observed in the temperature range of both transformations. (**Figures 6(a)**, B, and **6(b)**). In the case of the first transformation the effect was quite pronounced. This is thought to be evidence of a two-way shape memory effect in these alloys. This performance can be amplified by mechanical work hardening of the samples at 20ºC. During abrading of the 0.9 mm sample to the 0.5 mm sample it spontaneously began to bend at room temperature.

One possible explanation for the simple shape of the bending and recovery shape curves in the 20ºC–750ºC temperature range, while the resistance curves demonstrate two separate effects at 750ºC–650ºC and 350ºC–250ºC corresponding to two martensitic transformations, is that the electrical resistance measurement technique registers the occurrence of only thermal martensite whereas bending of the sample may be caused by the formation of both thermal and deformation martensites. The latter may appear above the M_s point as a result of bending load, as was observed in the case of alloys based on TiNi (13).

Thus despite the high temperature (~750ºC) the first martensitic transformation in Rh-0.1Sc-Ti alloy is accompanied by a shape memory effect when heated at a rate of about 20ºC min^{-1}. It has recently been reported that the ZrIr equiatomic compound with a temperature of martensitic transformation about 740ºC reveals a partial shape recovery, ~75%, only on a rapid heating of the sample (~100ºC sec^{-1}) (14).

Dilatometry

The results of dilatometric analysis of alloy with 0.1 at% Sc agree with those of the electrical resistance and thermomechanical tests. There are two inflections in
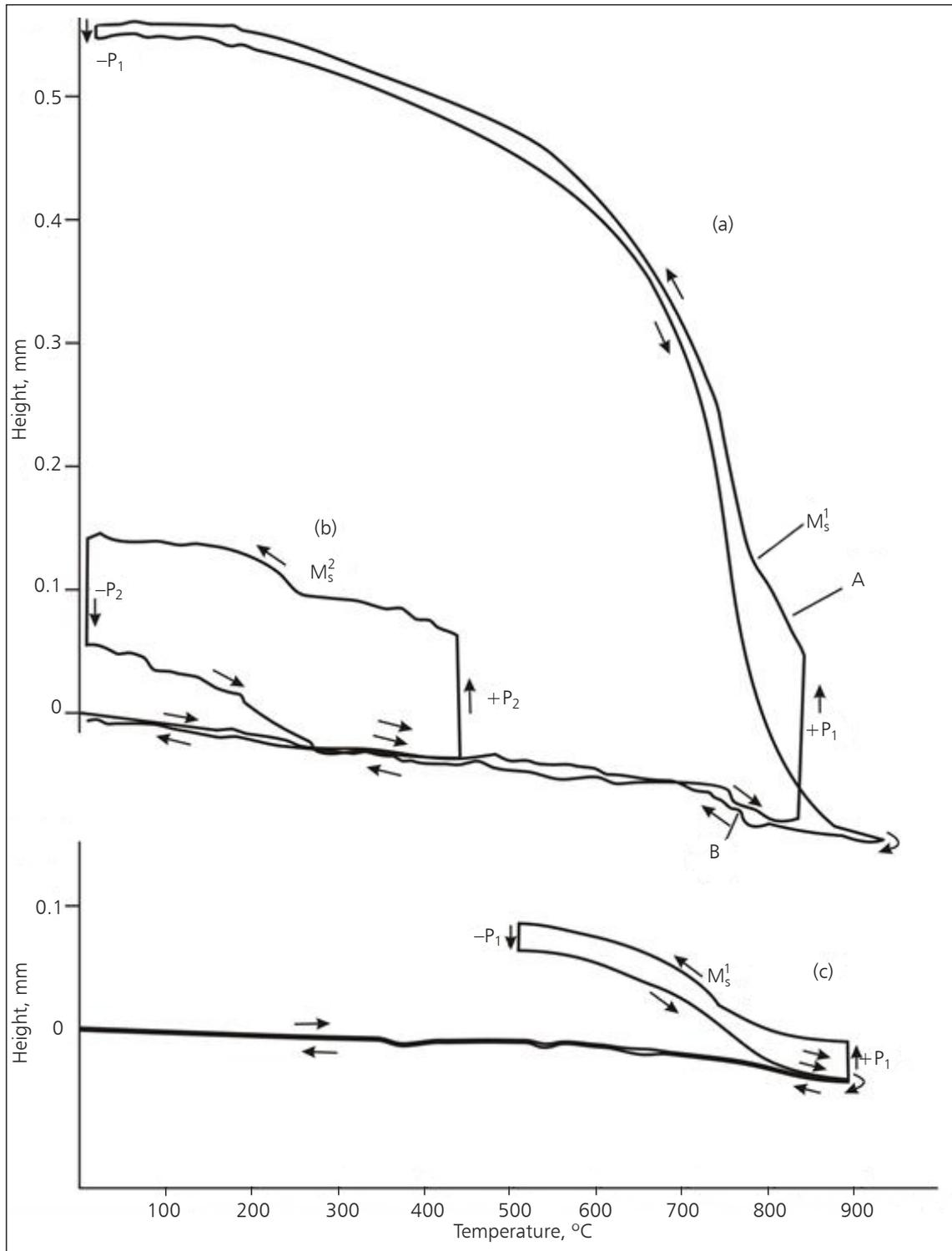
Fig. 6. Thermomechanical curve of the 50Rh-0.1Sc–Ti alloy: (a) full bending curve; (b) partial bending curve in the region of the second MT; (c) partial bending curve in the region of the first MT; A – area of deformation martensite prior to the first transformation; and B – a two-way shape memory effect

the dilatometric curve, corresponding to the beginning of the first (A) and second (B) MTs (**Figure 7**). A sharp change in the length of the sample in a narrow temperature range related to the first MT was observed as an abnormal effect on the electrical resistance curve at the same temperature for the alloys containing more scandium (**Figures 2(a)** and **2(b)**).

Ductility

It was noticed that the Rh-Sc-Ti alloys are only slightly oxidised at 20ºC–950ºC and are quite ductile at 900ºC. A sample of the alloy with 0.1 at% Sc having a section of 0.5 × 1.3 mm was bent at an angle of 90º, then was straightened and bent again in the opposite direction by 90º at 900ºC. It remained undestroyed and without any sign of cracks. In order to elucidate the degree of plasticity of the alloy, hot rolling of a 0.4 × 6 × 2 mm sample cut from ingot was performed. The sample was packed in an envelope made of stainless steel. Initial package thickness was 2.3 mm. As a result of 15 cycles of rolling and heating to 950ºC the thickness of the package decreased to 0.8 mm. The obtained sample was 0.18 mm thick and 17 mm long. From the experiment it follows that at 950ºC this alloy is ductile whereas at 20ºC it is not. Therefore, parts of heat sensitive items can be manufactured by hot rolling and extrusion. Not all alloys with a shape memory effect can be used for high-temperature materials; from observations on the mechanical properties of 50 at% Rh-0.1 at% Sc-Ti alloy, it can be assumed that material based on it would not meet difficulties in processing.

Conclusion

The transformation characteristics and recovery behaviour of ScRh-TiRh alloys have been studied. The results show that ScRh-TiRh alloys containing
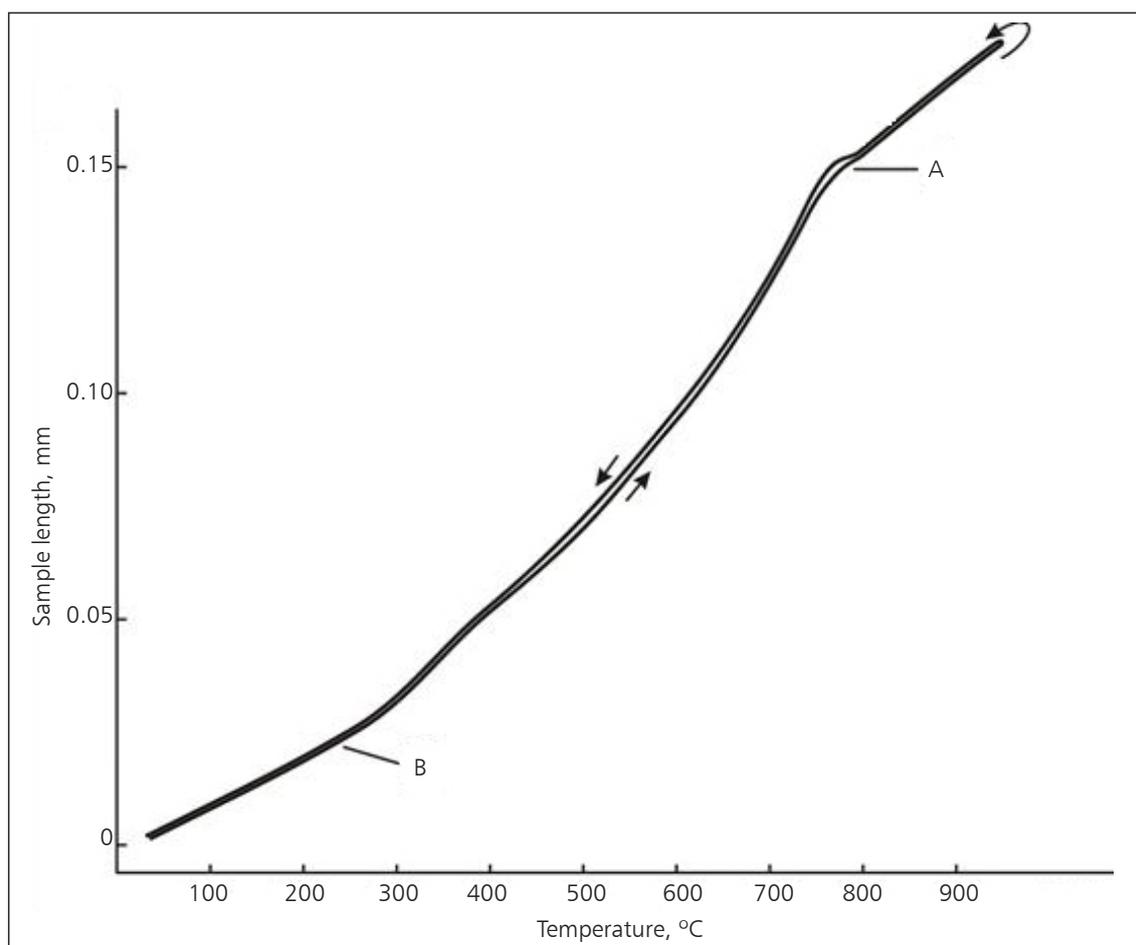

Fig. 7. Dilatometric curve of the 50Rh-0.1Sc-Ti alloy: A, B – effects related to transformation in the alloy (see text for explanation)

0–3.5 at% Sc undergo two high-temperature martensitic transformations: $M_s^1 \leq 750°C$ and $M_s^2 \leq 340°C$; while alloys with 16.7 at% and 21 at% Sc reveal only one MT. Both transformations are responsible for the shape memory effect in these alloys. The temperature of martensitic transformations in ScRh-TiRh alloys depends on the scandium content and decreases on substitution of scandium for titanium. The martensitic transformation temperatures of ScRh-TiRh alloys demonstrate low hysteresis. Transformations occurring in TiRh and ScRh-TiRh alloys are first order. TiRh binary and 50 at% Rh-Sc-Ti ternary alloys can display 100% shape memory effect in two temperature ranges up to 850°C; TiRh and ScRh-TiRh alloys also exhibit a two-way shape memory effect.

All characteristics of ScRh-TiRh alloys observed in the study make them good candidate materials for high-temperature shape memory alloys. Both TiRh and 50 at% Rh-Sc-Ti alloys display the unusual features of continuity of the deformation process on cooling and continuity of shape recovery process when heated in a temperature range from 20°C to 750°C, which might be due to two stages of transformation. The large range of shape memory effect observed in this work suggests that these alloys may be useful in different temperature-regulating elements that are designed to work in a wide temperature range.

This article presents the results of a preliminary investigation of the unusual properties of Rh-Sc-Ti alloys, in particular with respect to their thermomechanical behaviour. Further experimental work is required in order to confirm the effects observed. It is hoped that the article will be of interest to other scientists and will stimulate cooperation.

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