Anomalous temperature dependence of the yield strength in IVa–VIII intermetallic compounds with B2 structure

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The mechanical properties of the binary compound CoHf, and the pseudo-binary compounds CoTi–CoZr and CoTi–NiTi with B2 structure were measured as functions of testing temperature, composition and strain rate. In all of investigated compounds, the anomalous (positive) temperature dependence of the yield strength was observed at intermediate temperatures. The yield strength of CoHf compounds was dependent on composition through the contained second phase particles. The *pseudo*-binary compounds CoTi–CoZr were strengthened by a mutual solid solution of Ti and Zr, due to the atomic size difference between two atoms. The *pseudo*-binary compounds CoTi–NiTi were, by a mutual solid solution of Co and Ni, strengthened at low temperatures but softened at intermediate and high temperatures. A positive strain rate dependence of the yield strength was clearly found at high temperatures in CoHf compounds. Very high strain-hardening rates were observed at intermediate temperatures, usually accompanied by serrations on the stress–strain curves. Based on these results, it was proposed that the intermetallic compounds with B2 structure consisting of IVa and VIII transition metals universally exhibit the anomalous temperature dependence of the strength and therefore involve the similar deformation mechanism. Also, a possible deformation mechanism was considered in each temperature region.

1. Introduction

Among B2 (CsCl)-type intermetallic compounds which can form between various kinds of elements, the anomalous (i.e., positive) temperature dependence of the yield strength has been investigated for the Kurnakov-type compounds of β-CuZn [1] and FeCo [2]. These two B2 compounds exhibit order–disorder transition below their melting points. On the other hand, for Berthollide- and Daltonide-type compounds such as NiAl, FeAl and CoAl which remain ordered up to their melting point, the yield strength and flow strength have been shown to decrease monotonically with increasing temperature (a recent article reviews these [3]).

It was very recently shown that two compounds of CoTi [4] and CoZr [5] exhibited the anomalous temperature dependence of the yield strength in the polycrystalline forms; the yield strength increased from ambient temperature with increasing temperature, and then made a peak, followed by a rapid decrease at sufficiently high temperatures. In addition, the yield strength increased from ambient temperature with decreasing temperature. CoTi compound is the Berthollide-type while CoZr compound is considered to be the Daltonide-type. Both compounds consist of

IVa and VIII transition metals in a periodic table and are thus supposed to be strongly ordered. Also in the latest investigation using the single crystals of CoTi compounds [6], this kind of yield strength anomaly was observed and then attributed to the {1 1 0} ⟨0 0 1⟩ slip mode.

Co element also combines with Hf in IVa group and forms the B2 compounds. Therefore, it is expected that the similar strength anomaly and the similar deformation mechanism may be involved in this compound. Ductile–brittle transition temperature (DBTT) evaluated by the four point bend testing and steady state creep rate characterized by compression testing have been reported in CoHf compounds [7]. However, the yield strength properties of B2-type CoHf have not been reported so far.

In this work, the mechanical properties of CoHf compounds are represented firstly by compression test. Eventually, CoHf compounds are shown to exhibit the anomalous temperature dependence of the strength. This result reveals that B2-type compounds consisting of IVa and VIII group elements generally may exhibit the anomalous temperature dependence of the yield strength. Since Fe(VIII)Ti(IVa), Co(VIII)Ti(IVa), Co(VIII)Zr(IVa), Co(VIII)Hf(IVa)

and Ni(VIII)Ti(IVa) compounds indeed form B2-type structure, the *pseudo*-binary alloy systems composed of these compounds are expected to form the continuous solid solution field with a B2 structure. Here, the mechanical properties of Co(Ti, Zr) and (Co, Ni)Ti compounds among these *pseudo*-binary compounds are investigated for the purpose of improving the strength and toughness over their binary compounds.

The mechanical properties were measured by compression test and were then characterized by the yield strength and the plastic flow behaviour after yielding. The temperature dependence and strain rate effect (as the environmental factor) and the compositional effect (as the material factor) were also investigated for these binary and ternary compounds.

2. Experimental procedure

The compounds used in this work were prepared by non-consumable arc melting under an argon gas, using cobalt of 99.9 wt % purity, hafnium of 95 wt % purity (most of the remaining element is zirconium), titanium of 99.9 wt % purity, zirconium of 99.6 wt % purity and nickel of 99.9 wt % purity. The buttons of about 120 g were homogenized in a vacuum at 1223 K for 34 h, followed by furnace cooling. The analysis of chemical compositions was not carried out because the weight loss during melting and homogenizing was very little. Compression samples, approximately 2×2 mm^2 in cross-section and 5 mm length, were cut from the homogenized buttons by a precision wheel cutter. Here, the specimen axis was taken parallel to the axis of the columnar grains with a grain size of a few hundred micrometres. These microstructures were usually developed from bottom to top of the button during solidification.

Compression tests were conducted on an Instron-type machine at an initial strain rate of 3.3×10^{-4} s^{-1} (and also 3.3×10^{-3} s^{-1} in order to see the strain rate effect) at temperatures from 77 to 1073 K. The tests at 77 K were performed in liquid nitrogen while the tests at remaining temperatures were performed under a vacuum of about 1.3×10^{-3} Pa. Samples were mostly deformed to about a few per cent strain. However, a few samples fractured at even smaller strains before the macroscopic yielding.

Metallographic observation and X-ray diffraction measurement were done for plate-like samples cut from the homogenized buttons. X-ray diffraction was measured at an accelerated voltage of 35 kV by using CuK_α radiation.

3. Results
3.1. Microstructures
3.1.1. CoHf compounds
This compound is reported to have a melting point of 1913 K and very limited solid solution range as shown in Fig. 1 [8]. To confirm the solid solubility of a single phase with B2 structure, the compounds with a number of alloy compositions were examined. Both metallographic observation and X-ray diffraction indicated second phase particles in all of prepared alloy
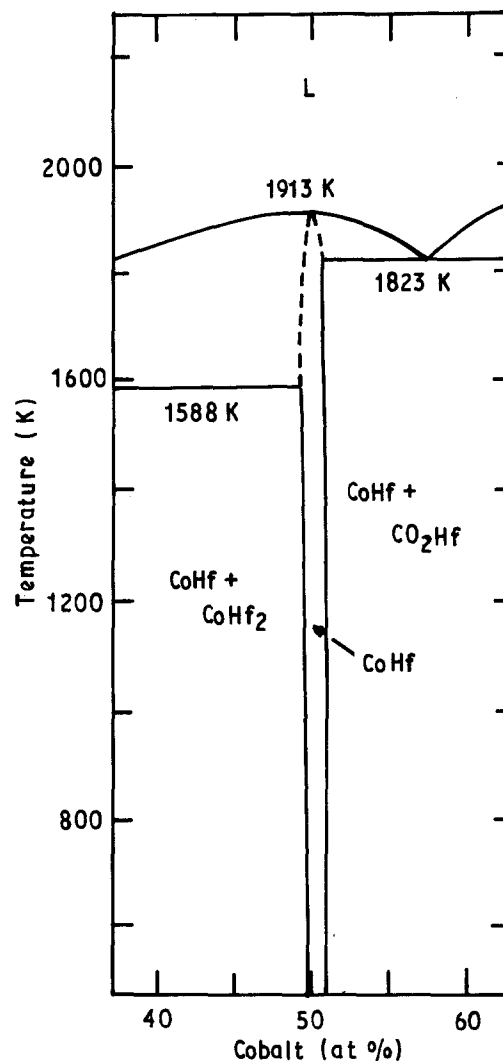

Figure 1 Phase diagram near the *equi*-atomic composition on the Co–Hf system [8].

compositions. Just-stoichiometric compound also contained CoHf$_2$ and Co$_2$Hf particles, suggesting that the compound was not in an equilibrium condition. Figs 2 and 3 show the microstructures and the results of X-ray diffraction of two alloy compositions, i.e., slightly Hf-rich composition (50.3 at %Hf) and slightly Co-rich composition (49.5 at %Hf), respectively. Fig. 2 clearly shows the second phase particles, the morphology of which exhibits the structures formed by the dendritic growth during solidification. Also, it was shown by X-ray diffraction that the former compound contained CoHf$_2$ while the latter compound contained both CoHf$_2$ and Co$_2$Hf. Also, it is noted in Fig. 3 that the peaks which could not be identified as B2, CoHf$_2$ or Co$_2$Hf structures were also observed.

The lattice parameter of CoHf compounds was shown to be 0.31677 nm in all of the prepared alloy compositions. This result reveals that a single phase of CoHf compound with B2 structure is limited to more than that reported in Fig. 1 [8]. Therefore, this compound may be categorized as the Daltonide-type rather than as the Berthollide-type. Determined lattice parameters were almost identical to the values (0.3165 nm [9] and 0.3164 nm [10]) previously reported.
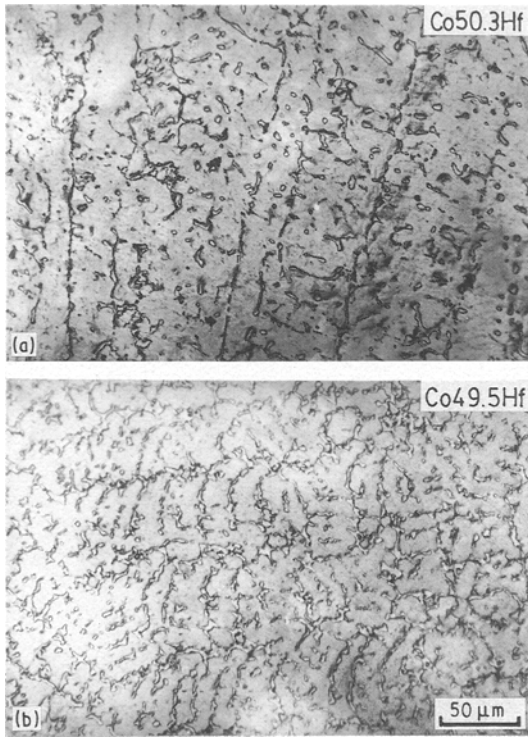
Figure 2 Microstructures of (a) slightly Hf-rich composition (50.3 at %Hf) and (b) slightly Hf-poor composition (49.5 at %Hf).

Co(Ti, 15 at %Zr) along the *pseudo*-binary line between CoTi and CoZr. Metallographic observation in these samples showed a matrix phase and trace amount of second phase particles. The second phase particles occasionally delineated the grain boundaries. However, these particles were not identified because the peaks from these particles in X-ray diffraction were too weak to detect. Fig. 5 shows the change of the lattice parameters with Zr content; the lattice parameters increased linearly with increasing Zr content. This result implies that the continuous solid solution of B2 structure developed to the limiting composition of CoZr, and also Ti elements in CoTi were substituted by Zr elements.

It was shown in a study of the *pseudo*-binary system FeTi–CoTi–NiTi that the complete solid solutions with B2 structure were formed [11]. Fig. 4b shows the prepared alloy compositions i.e., (Co, 5 at %Ni)Ti and (Co, 8 at %Ni)Ti along *pseudo*-binary line between CoTi and NiTi. These compounds also contained a trace amount of second phase particles which preferentially delineated along the grain boundaries. Fig. 5 shows the change of the lattice parameters with Ni content; the lattice parameters increased linearly with increasing Ni content, thus being consistent with the previously reported results [11].

3.1.2. Co (Ti, Zr) and (Co, Ni)Ti compounds

Since Co forms the same B2 structure combined with Ti and Zr, it is expected that the continuous solid solution of B2 structure exists along the *pseudo*-binary line between CoTi and CoZr although the phase diagram supporting this has not been available.

Fig. 4a shows the prepared alloy compositions, i.e., Co(Ti, 5 at %Zr), Co(Ti, 10 at %Zr) and

3.2. Mechanical properties
3.2.1. CoHf compounds

The temperature dependence of the 0.2% yield strength of CoHf compounds containing 50.3 at %Hf and 49.5 at %Hf, which were measured at two strain rates of 3.3×10^{-4} s^{-1} and 3.3×10^{-3} s^{-1}, is shown in Fig. 6. Three distinct temperature regions divided
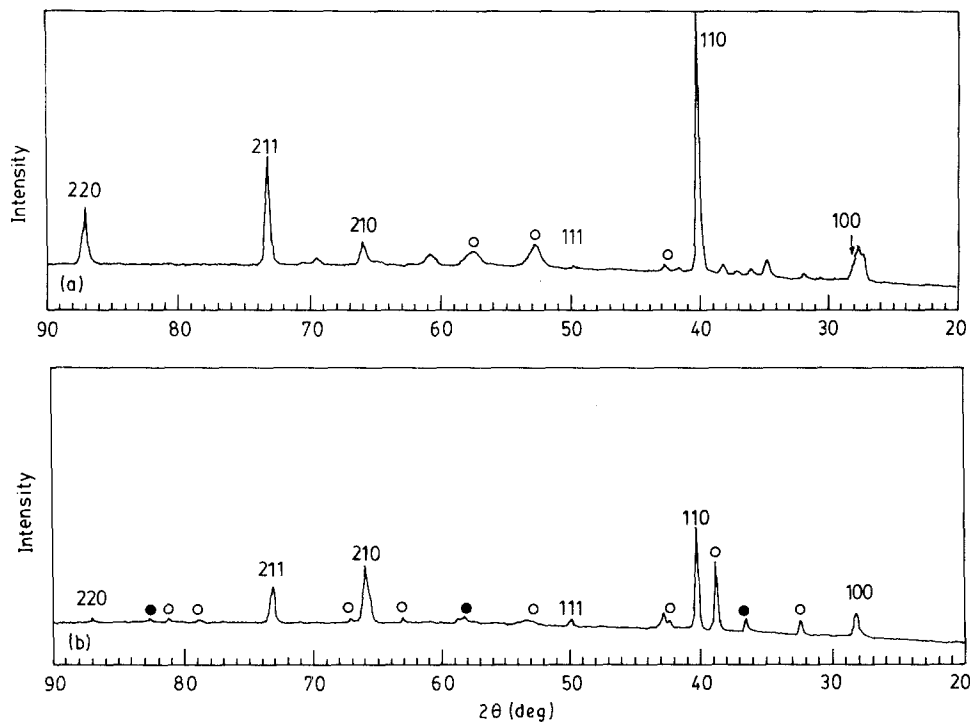

Figure 3 X-ray diffraction patterns of (a) slightly Hf-rich composition (50.3 at %Hf) and (b) slightly Hf-poor composition (49.5 at %Hf); (○) CoHf$_2$, (●) Co$_2$Hf.
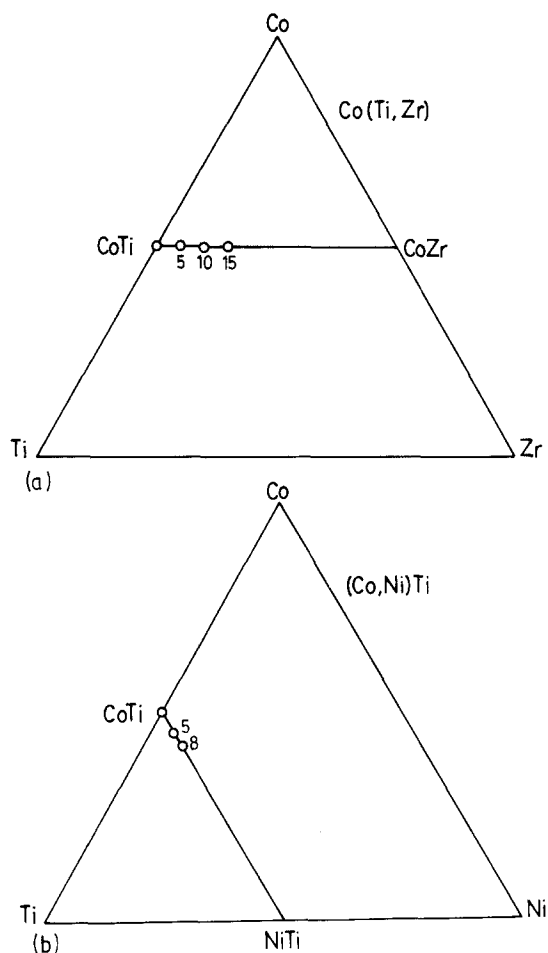
Figure 4 Representation of compositions of (a) Co(Ti, Zr) and (b) (Co, Ni)Ti compounds prepared in this work.
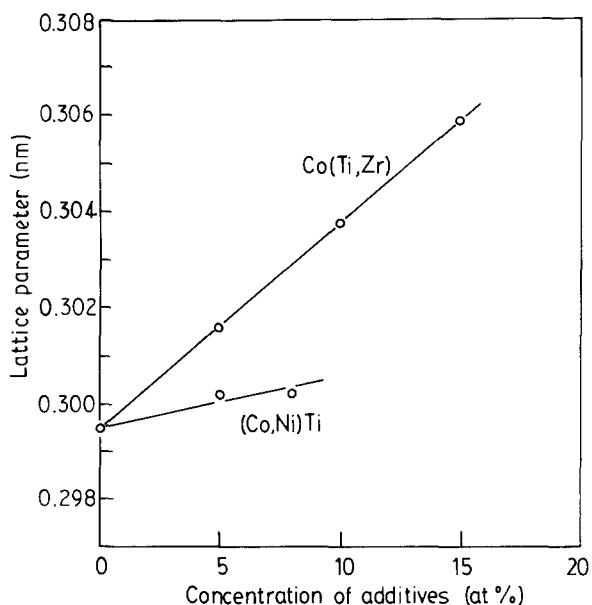

Figure 5 Changes in the lattice parameters with Zr content in Co(Ti, Zr) compounds and with Ni content in (Co, Ni)Ti compounds, respectively.

by the bottom temperature (T_b) and the peak temperature (T_p) of the yield strength were distinguished as has been observed in polycrystals [4] and single crystals [6] of CoTi compounds, and polycrystals of CoZr compounds [5].
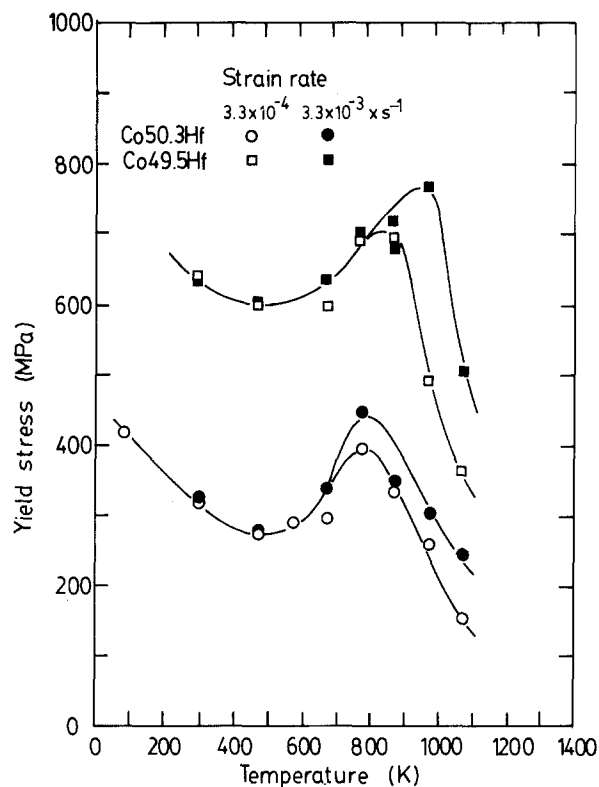

Figure 6 Temperature and composition dependence of the yield strength of CoHf compounds, measured at strain rates of 3.3 × 10^{-4} s^{-1} and 3.3 × 10^{-3} s^{-1}.

(i) Low temperature region: the yield strength increased with decreasing temperature from T_b.

(ii) Intermediate temperature region: the yield strength increased steeply with increasing temperature from T_b.

(iii) High temperature region: the yield strength decreased rapidly with increasing temperature from T_p.

The compositional effect on the temperature–yield strength curves shows that lower Hf content resulted in higher values of the yield strength at almost whole test temperatures. In this figure, it appears that the strength level in two compositions is attributed to the difference in the morphology and density of the second phase particles as shown in Fig. 2 and identified by Fig. 3. On the other hand, the change of the yield strength with temperature is not affected by the second phase particles but by the matrix phase of B2 structure. The peak temperature, T_p, showed slightly higher temperature in compounds containing 49.5 at %Hf.

It is interesting to note that T_p observed in Fig. 6 is basically identical to the DBTT measured by the four point bend testing [7]. Therefore, it may be pointed out that the deformation mode at high temperatures is inherently ductile in contrast to that at temperatures below T_p.

Concerning the strain rate effect on the yield strength, Fig. 6 exhibits that a positive strain rate dependence is obvious at temperatures above T_p and negligibly small at temperatures below T_p.

The nature of plastic flow of CoHf compounds is shown in Fig. 7, where the stress–strain curves (the load–displacement curves if strictly describing) of the
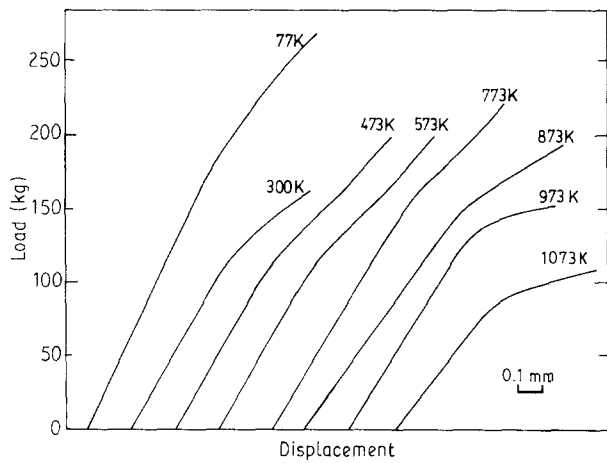
Figure 7 Stress–strain curves of CoHf compounds containing 50.3 at %Hf which were deformed at various temperatures and measured at a strain rate of $3.3 \times 10^{-4}\,\text{s}^{-1}$.

compounds containing 50.3 at %Hf were, as a representative, displayed at various temperatures and at a strain rate of $3.3 \times 10^{-4}\,\text{s}^{-1}$. The stress–strain curves after yielding were smooth at whole test temperatures in contrast to polycrystals [4] and single crystals [6] of CoTi compounds. In CoTi compounds, the jerky flow (the serrations) occurred at intermediate temperatures between T_b and T_p. Also, it must be noted that the steady state flow was established at temperatures above T_p.

Strain-hardening rate after yielding was also evaluated from the difference in the flow strength levels between 1 and 0.2% plastic strains (i.e., $\Delta\sigma = \sigma_{1\%} - \sigma_{0.2\%}$). Fig. 8 shows the temperature dependence of the strain-hardening rate of the compounds containing 50.3 at %Hf which were measured at two strain rates (of $3.3 \times 10^{-4}\,\text{s}^{-1}$ and $3.3 \times 10^{-3}\,\text{s}^{-1}$). $\Delta\sigma$ shows the maximum (peak) approximately at a temperature where the maximum in the yield strength–temperature curve appeared (Fig. 6). Here, it
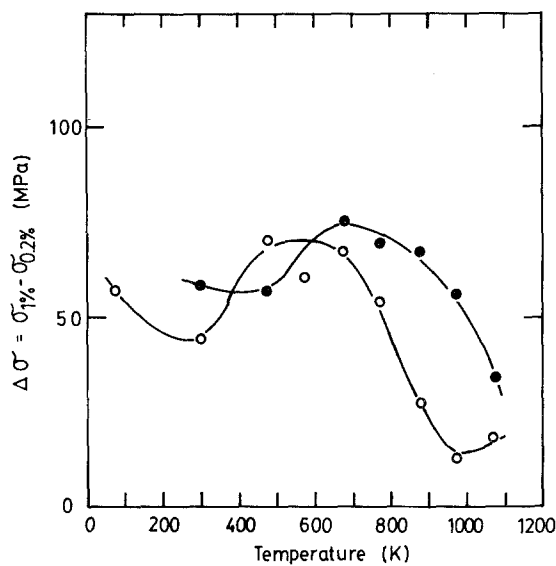
is strongly suggested that particularly at both intermediate and high temperatures, there is a tight correlation between the yield strength and the strain-hardening behaviour. Thus, higher yield strength accompanied with higher strain-hardening rate after yielding.

3.2.2. Co(Ti, Zr) and (Co, Ni)Ti compounds

The temperature dependence of the 0.2% yield strength of the *pseudo*-binary compounds CoTi–CoZr, which were measured at a strain rate of $3.3 \times 10^{-4}\,\text{s}^{-1}$, is shown in Fig. 9. Here, the data for pure CoTi compounds [4] were also included in this figure in comparison. Co(Ti, 15 at %Zr) compounds were not fabricated into compression samples because of their extreme brittleness. Also, it is noted in this figure that the Co(Ti, 10 at %Zr) compounds tested at temperatures below T_p fractured before macroscopic yielding and therefore reliable data were not plotted. The yield strength of these ternary compounds showed the similar temperature dependence to those of CoTi [4] and CoZr [5] compounds as expected, and also to CoHf compounds observed in the previous section. Regarding the compositional effect on the yield strength and their temperature dependence, the addition of Zr element considerably strengthened CoTi compounds at whole test temperatures and also slightly increased the peak temperature, T_p. This strengthening appears to be due to a large difference in the atomic size between Ti and Zr elements, as evaluated from the lattice parameter change in Fig. 5.

The stress–strain curves of Co(Ti, 5 at %Zr) compounds which were deformed at various temperatures
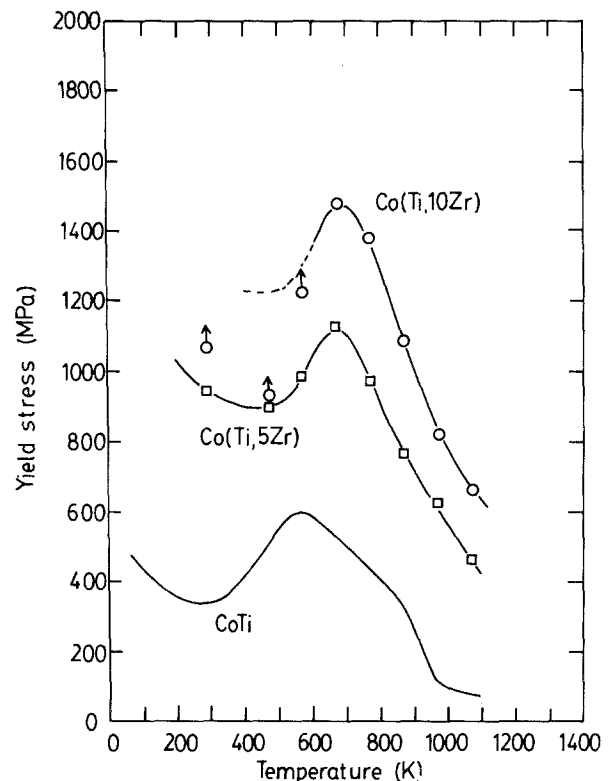

Figure 8 Temperature and strain rate dependence of the strain-hardening rate of CoHf compounds containing 50.3 at %Hf; at strain rates of (○) $3.3 \times 10^{-4}\,\text{s}^{-1}$, (●) $3.3 \times 10^{-3}\,\text{s}^{-1}$. Note that the strain-hardening rate was evaluated by $\Delta\sigma = \sigma_{1\%} - \sigma_{0.2\%}$.

Figure 9 Temperature and composition dependence of the yield strength of Co(Ti, Zr) compounds, measured at a strain rate of $3.3 \times 10^{-4}\,\text{s}^{-1}$.

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and at a strain rate of $3.3 \times 10^{-4} \, s^{-1}$ are shown in Fig. 10. The stress–strain curves were serrated at intermediate temperatures between T_b and T_p. This result is consistent with those in CoTi compounds [4] but inconsistent to those in CoHf compounds observed in this work. Strain-hardening rate (evaluated by $\Delta\sigma = \sigma_{1\%} - \sigma_{0.2\%}$) in Fig. 11 again showed the maximum (peak) approximately at a temperature where the maximum in the yield strength–temperature curve (Fig. 9) occurred. Thus, higher yield strength accompanied with higher strain-hardening rate after yielding, again suggests that there is a tight correlation between the yield strength and the strain-hardening behaviour.

Fig. 12 exhibits the temperature dependence of the 0.2% yield strength of the *pseudo*-binary compounds CoTi–NiTi, which were measured at a strain rate of $3.3 \times 10^{-4} \, s^{-1}$. These ternary compounds also exhibited the anomalous temperature dependence of the

Figure 12 Temperature and composition dependence of the yield strength of (Co, Ni)Ti compounds, measured at a strain rate of $3.3 \times 10^{-4} \, s^{-1}$.

yield strength, being similar to a number of the compounds previously observed. However, the strength levels of (Co, Ni)Ti compounds were rather enhanced at low temperatures, below T_b, but were reduced at intermediate and high temperatures, above T_b. This result reveals that the alloying of Ni into CoTi compounds affected in different ways the strength behaviour at these temperature regions.

Fig. 13 summarizes the stress–strain curves of the *pseudo*-binary compounds CoTi–NiTi which were deformed at various temperatures and at a strain rate of $3.3 \times 10^{-4} \, s^{-1}$. The stress–strain curves were serrated at intermediate temperatures between T_b and T_p similarly to Co(Ti, Zr) compounds. Fig. 14 plots the strain-hardening rate, $\Delta\sigma$, of (Co, Ni)Ti compounds. Here, it is again noted that $\Delta\sigma$ showed the maximum
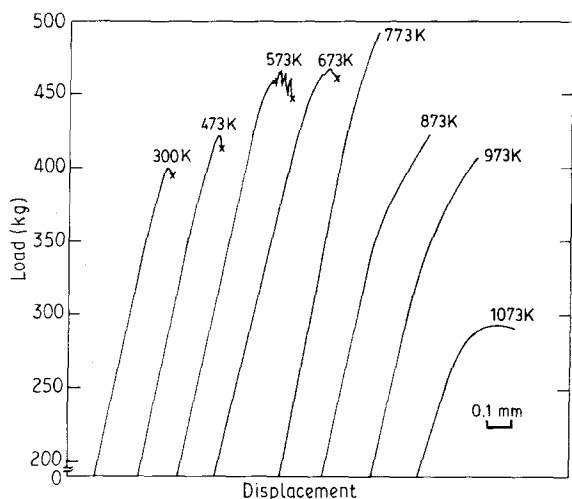

Figure 10 Stress–strain curves of Co(Ti, 5 at %Zr) compounds which were deformed at various temperatures and measured at a strain rate of $3.3 \times 10^{-4} \, s^{-1}$.

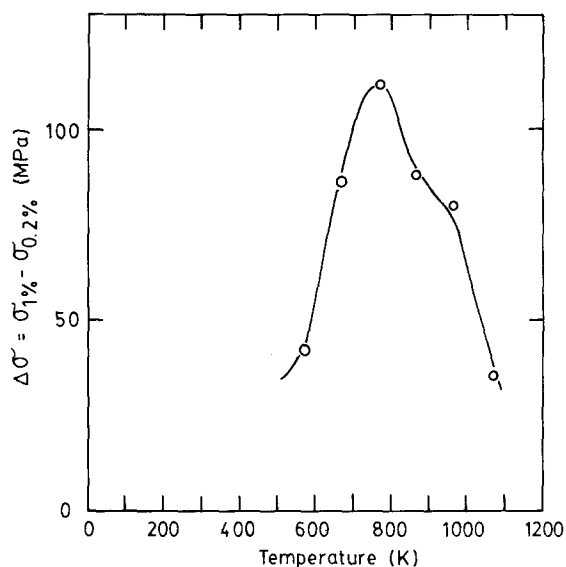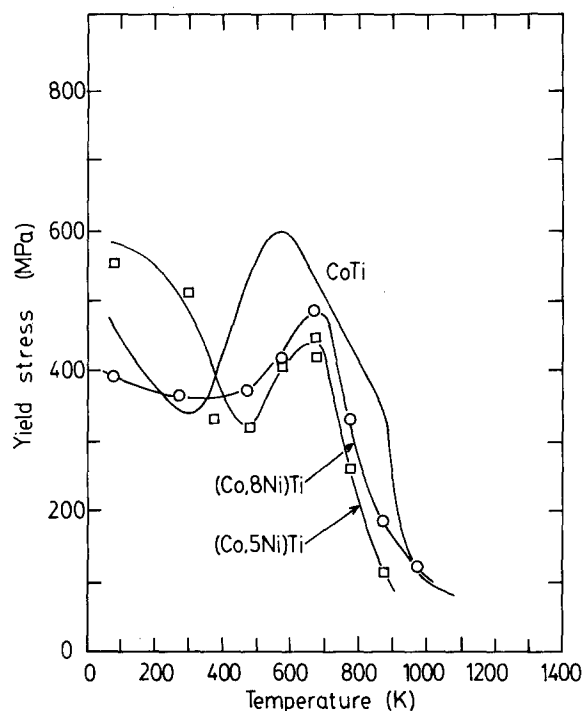*Figure 11* Temperature dependence of the strain-hardening rate of Co(Ti, 5 at %Zr) compounds, measured at a strain rate of $3.3 \times 10^{-4} \, s^{-1}$. Note that the strain-hardening rate was evaluated by $\Delta\sigma = \sigma_{1\%} - \sigma_{0.2\%}$.
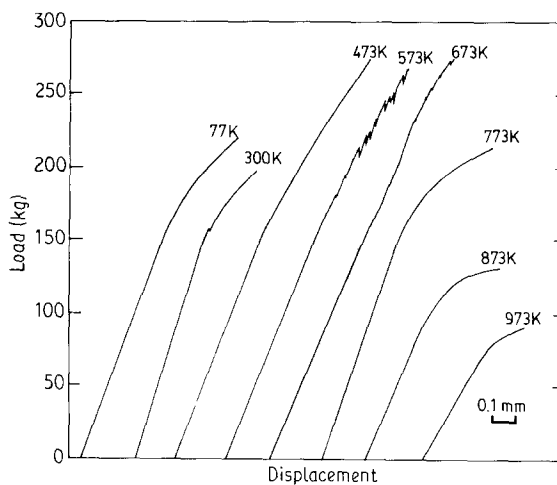

Figure 13 Stress–strain curves of (Co, 8 at %Ni)Ti compounds which were deformed at various temperatures and measured at a strain rate of $3.3 \times 10^{-4} \, s^{-1}$.
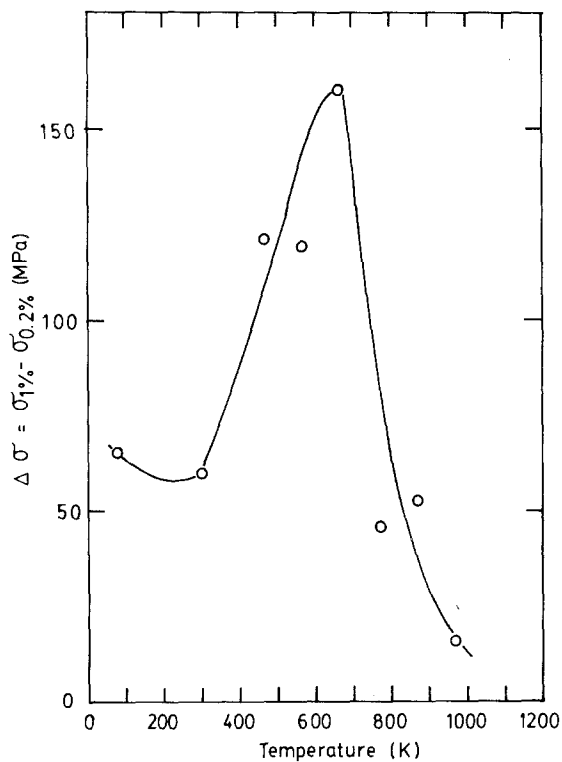
Figure 14 Temperature dependence of the strain-hardening rate of (Co, 8 at %Ni)Ti compounds, measured at a strain rate of 3.3 × 10^{-4} s^{-1}. Note that the strain-hardening rate was evaluated by $\Delta\sigma = \sigma_{1\%} - \sigma_{0.2\%}$.

(peak) approximately at a temperature where the maximum in the yield strength–temperature curve (Fig. 12) occurred.

4. Discussion

B2 structures which consist of IVa and VIII transition metals, FeTi, CoTi, CoZr, CoHf and NiTi compounds have been reported. Among these compounds, CoTi [4, 6] and CoZr [5] compounds have been observed to show the anomalous (positive) temperature dependence of the yield strength. In this work, CoHf compounds were newly found to show the similar temperature dependence of the strength. Also, it was very recently known that NiTi compounds, B2 structure of which was stabilized by addition of Co and Cr elements showed the similar anomalous temperature dependence of the strength [12]. Therefore, it may be described that IVa–VIII intermetallic compounds with B2 structure universally exhibit the anomalous, positive temperature dependence of the yield strength although the strength property of FeTi compounds has not been reported yet. Also, it is expected that the *pseudo*-binary compounds CoTi–CoZr–CoHf and FeTi–CoTi–NiTi involve the similar deformation mode at a wide range of temperatures. Indeed, the *pseudo*-binary compounds CoTi–CoZr, i.e., Co(Ti, Zr) and CoTi–NiTi, i.e., (Co, Ni)Ti conserved the similar temperature dependence of the yield strength.

These results suggest that the alloy design to control the strength and the ductility is possible in these *pseudo*-binary compounds. Flow strength may be enhanced by addition of larger or smaller sized

atoms into the binary compounds, e.g., CoTi–CoZr compounds described in this work. On the other hand, it has been shown by Scholl that the ductility (toughness) at room temperature increased steadily with increasing electron concentration (e/a), e.g., in the order of FeTi, CoTi and NiTi compounds [11]. This increase has been interpreted as resulting from an increase in the metallic nature of the bonding, from directional A–B bonds in FeTi compounds to a more metallic (nondirectional) type of the bonding in NiTi compounds. Also, it is likely that the ductility decreases with increasing atomic size of the component atom of IVa, e.g., in the order of CoTi, CoZr and CoHf compounds.

The variation of the strength with temperature of strongly-ordered B2 compounds was generally divided into three regions (four regions if that at very high temperatures is included although this region has not been explicitly recognized). At low temperatures, below room temperature (region (i)), the yield strength was strongly dependent on temperature and increases rapidly with decreasing temperature. This has been attributed to being controlled by the Peierls double kink mechanism.

At intermediate temperatures beyond room temperature, the most striking result has been observed; in B2 compounds consisting of VIII and IIIb elements like FeAl, CoAl and NiAl, the yield strength remained approximately independent of temperature or slowly decreased with increasing temperature, while in B2 compounds consisting of IVa and VIII elements, the yield strength increased rapidly with increasing temperature as observed in this work. Many deformation modes have been proposed at these temperatures of the former B2 compounds. Impurity atoms have a strong effect on yielding, producing the peak due to phenomena such as the Portevin–Le Chatelier effect. Also, the change of slip systems with temperature observed in some compounds produced the peak of the yield strength, for example, FeAl compounds [13]. On the other hand, it was shown that in single crystals of CoTi compounds consisting of IVa–VIII elements, deformation occurred by slip of {1 1 0} ⟨0 0 1⟩ and was very much dependent on the sample orientation [6]. This result reveals that the deformation at this temperature is attributed neither to the interaction of impurity (or solute) atoms with dislocations nor to the change of slip systems. Two possibilities were, alternatively, proposed to explain the anomalous positive temperature dependence of the strength observed in this compound [6]. One is that a ⟨0 0 1⟩ superdislocation decomposes into (some) partials connected with plane fault such as stacking fault or anti-phase boundary, or climb-dissociates on the non-slip plane. Either case was postulated to introduce an increase of the flow strength at intermediate temperatures. However, it was not certain what kind of locking or pinning is responsible for the anomalous temperature dependence of the strength [6].

At high temperatures, deformation in strongly ordered B2 compounds was controlled by slip. Also in the case of single crystals of CoTi compounds [6], slip dominated the deformation although slip vector

was not certain [6]. The strongly negative temperature dependence of the strength at high temperatures has been generally interpreted by a mechanism similar to that at low temperatures, i.e., the Peierls mechanism. This deformation mode was generally strain rate dependent. The compounds observed in this work indeed exhibited strong strain rate dependence on the yield strength at this temperature region.

Finally, the jerky flow (i.e., the serration in the stress–strain curves) observed at intermediate temperatures in Co(Ti, Zr) and (Co, Ni)Ti compounds is discussed (Figs 10 and 13). This feature was also observed in single crystals of CoTi compounds when their sample orientations are close to [0 0 1], and then attributed to the introduction of kinkings. As the reason why kinkings were introduced, it was proposed that the resolved shear stress (RSS) on a favourable slip direction (i.e., [0 0 1]) of this compound was too low in this sample orientation. Compression samples of Co(Ti, Zr) and (Co, Ni)Ti compounds were taken parallel to the columnar grains and therefore the axes of the majority of grains in the sample are supposed to have [0 0 1] orientations which are preferable growth direction of this kind of compound. Consequently, the kink deformation was introduced in these compounds, causing the serrations in the stress–strain curves. On the other hand, in the case of CoHf compounds, it appears that a relatively large amount of dendritic second phases prohibited preferential crystal growth of [0 0 1] (see Fig. 3) and therefore did not display the serrated flow.

5. Conclusions

The mechanical properties of the binary compound CoHf, and the *pseudo*-binary compounds CoTi–CoZr and CoTi–NiTi with B2 structure were measured by compression test. The tests were performed as functions of testing temperature, composition and strain rate. The results obtained are as follows.

1. A single phase of CoHf was limited almost at stoichiometric composition. CoTi formed a continuous solid solution with CoZr and with NiTi, respectively.

2. In all of investigated compounds, three distinct regions were distinguished in the change of the yield strength with temperature; a low temperature region where the yield strength increased rapidly with decreasing temperature from T_b (bottom temperature), an intermediate temperature region where the yield strength increased with increasing temperature from T_b and a high temperature region where the yield strength again decreased rapidly with increasing temperature from T_p (peak temperature).

3. The yield strength of CoHf compounds was dependent on composition through the contained second phase particles. Solid solution between Ti and Zr in the *pseudo*-binary compounds CoTi–CoZr

created remarkable strengthening due to the difference in the atomic size of the two atoms. Solid solution between Co and Ni in the *pseudo*-binary compounds CoTi–NiTi introduced the strengthening at low temperatures but softening at intermediate and high temperatures.

4. A positive strain rate dependence of the yield strength was clearly found at high temperatures above T_p in CoHf compounds.

5. Very high strain-hardening rates were observed at intermediate temperatures, usually accompanied by the load drop (i.e., the serrations on the stress–strain curves).

6. Based on these observations, it was proposed that the intermetallic compounds with B2 structure consisting of IVa and VIII transition metals universally exhibit the anomalous temperature dependence of the strength and therefore involve the similar deformation mechanism. Also, it was suggested that the alloy design at the same time involving the strength and the ductility is possible in these *pseudo*-binary compounds with B2 structure.

Acknowledgement

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