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The effect of quenching on the martensitic transformation in β_1-phase of Au–Cd alloys

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The effect of quenching on the martensitic transformation mechanism in β_1 Au–Cd alloys has been investigated by measurements of the electrical resistivity and X-ray diffraction. In the case of the Au–47.5 at%Cd alloy, the ζ_2'-martensite is the characteristic product under quenching conditions, but it always exists with the equilibrium γ_2'-martensite phase. Consequently, the $\beta_1 \leftrightarrow \gamma_2'$ and $\beta_1 \leftrightarrow \zeta_2'$ transformations occur simultaneously during the heating and cooling cycles. The corresponding resistivity behaviour is very complicated and extremely sensitive to thermal treatments such as quenching temperature and thermal cycling. On the other hand, in the case of the Au–49.0 at%Cd alloy, only the $\beta_1 \leftrightarrow \zeta_2'$ transformation occurs even when quenched, and the transformation is unaffected structurally by quenching. A distinct resistivity anomaly, which is considered to be due to the disappearance of quenched-in vacancies, is observed in quenched alloys. Some important characteristics of this anomaly are determined. In particular, the quenching effect disappears when the specimen is heated above the temperature at which the resisitivity anomaly begins. This result suggests that the quenched-in vacancies play an essential role in the martensitic transformation process under quenching conditions.

1. Introduction

It is well known that the β_1-phase of Au–Cd alloys undergoes the martensitic transformation even during slow cooling. Until now, many investigations of the crystal structures of martensites in Au–Cd alloys have been made using various experimental techniques such as optical microscopy, electron microscopy, and diffraction and X-ray analysis [1–8]. The results obtained from these studies are very complicated, and in detail are inconsistent with each other. From the results, however, we can derive the following conclusion: the orthorhombic γ_2'-(modified 2H) and trigonal (or hexagonal) ζ_2'-martensites exist as equilibrium phases in the alloys with compositions of nearly 47.5 and 49.0 at%Cd, respectively. It has also been shown that transformations from the β_1-phase to the γ_2'- and ζ_2'-martensites cause characteristic electrical resistivity changes. Namely, the $\beta_1 \rightarrow \gamma_2'$ transformation exhibits a decrease in resistivity, while the $\beta_1 \rightarrow \zeta_2'$ transformation exhibits an increase in resistivity [9].

Another interesting feature of the transformation in Au–Cd alloys is the effect of quenching. For instance, in the case of the Au–47.5 at%Cd alloy, an increase in resistivity occurs through the transformation to the martensite, when quenched from high temperatures to just above or below the $\beta_1 \rightarrow \gamma_2'$ transformation point [10, 11]. That is, the resistivity change is contrary to the case of slow cooling. This behaviour has been attributed to the fact that quenching alters the transformation characteristic from the $\beta_1 \leftrightarrow \gamma_2'$ type to the $\beta_1 \leftrightarrow \zeta_2'$ one. Such a consideration, however, is based on the analogy of the resistivity behaviour in the $\beta_1 \rightarrow \zeta_2'$ transformation of the slow cooled Au–49.0 at%Cd alloy.

On the other hand, in the case of the Au–49.0

at%Cd alloy the resistivity behaviour is not significantly modified by quenching. According to Wechsler and Read [10], except for lowering the transformation temperature to the martensite by several degrees, the resistivity behaviour is almost the same as that for the slow cooled case; an increase in resistivity occurs through the transformation to the martensite in both cases of slow cooling and quenching. This result suggests that the $\beta_1 \leftrightarrow \zeta_2'$ transformation mechanism is practically unaffected by quenching.

Notwithstanding these studies, the transformation behaviours under quenching conditions are still not well understood as no detailed and/or systematic work on the effect of quenching has been undertaken for a wide range of compositions. In the present paper, we will interpret the effect of quenching on the characteristics of the martensitic transformation, based on the results obtained by measurements of electrical resistivity and X-ray diffraction for alloys containing 47.5 to 49.5 at%Cd.

2. Experimental procedure

Eight $Au_{100-x}Cd_x$ ($47.5 \leqslant x \leqslant 49.5$) alloys were used in the present study. They were prepared by melting preweighed Au (99.999%) and Cd (99.9999%) metals together in sealed quartz tubes filled with argon and shaking vigorously several times in order to ensure homogeneity. Alloy ingots were cut into some strips of sheet 0.4 mm thick for resistivity measurements. The resistivity curves were obtained by using a conventional potentiometric method. X-ray diffraction measurements were carried out for the Au–47.5 and 49.0 at%Cd alloys using both single crystals and powder specimens. The single crystals were grown by the Bridgman technique and cut into rectangular blocks (~5 mm x 7 mm x 10 mm) with flat surfaces parallel to the $(1\,1\,0)_{\beta_1}$ or $(1\,0\,0)_{\beta_1}$ plane. The crystal orientations were determined by the Laue back-reflection method at about 373 K, at which the crystals were in the β_1-phase. Powder specimens were prepared from polycrystals by filing until they were fine enough to pass through a sieve of 200 mesh. X-ray diffraction profiles were taken by a temperature-controlled goniometer with CuKα radiation under a vaccum atmosphere.

Just before the measurements, the specimens were annealed at 723 K for 30 to 60 min in a glass tube filled with argon and subsequently quenched in ice-water in which the glass tube immediately broke. For the Au–49.0 at%Cd alloy, in addition, the quenching was carried out from several temperatures in the range 812 to 627 K. All the measurements were begun at room temperature and then thermal cycles were performed with heating and cooling rates of about 2 K min^{-1}.

3. Results

The results of electrical resistivity measurements on the quenched Au–47.5 at%Cd alloy are shown in Fig. 1. Four thermal cycles were performed successively. The heating range was increased from the initial temperature to 361 to 388 K as indicated by the arrows. Fig. 1a shows a slight increase in resistivity occurring at about 332 K on the initial heating process. During cooling, the transformation occurs with an increase in resistivity and, by reheating, the resistivity curves exhibit a small transformation hysteresis. This characteristic remains unchanged if the maximum temperature

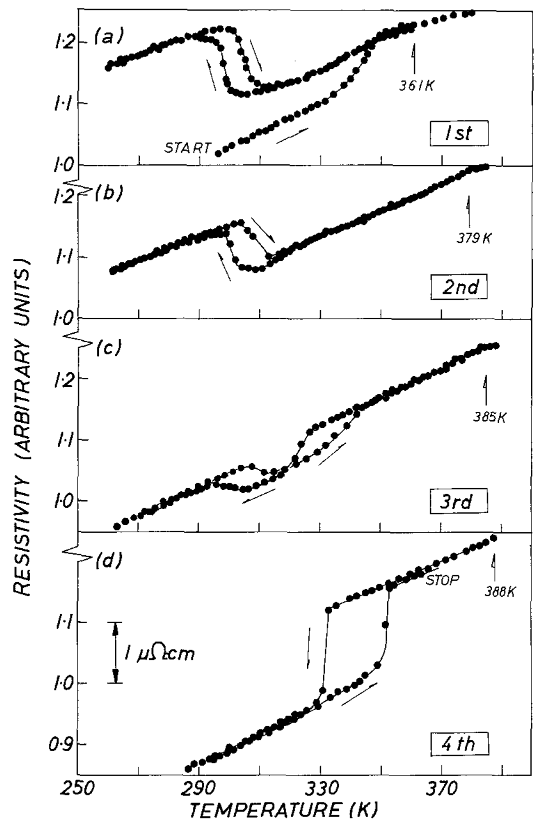

Figure 1 Resistivity against temperature curves for the quenched Au–47.5 at%Cd alloy on successive thermal cycles. Vertical arrows indicate the maximum heating temperature in each thermal cycle.

of heating is below about 380 K (Fig. 1b). The shape of the hysteresis loop is nearly the same as that found in slow-cooled Au–49.0 at%Cd alloy. After heating to about 385 K, the resistivity exhibits wave-like behaviour in a wide range of cooling and heating temperatures (Fig. 1c). Once the specimen is heated above about 388 K, the quenching effect disappears, i.e. the resistivity decreases through the transformation to the martensite on cooling (Fig. 1d).

Regarding the mechanism of the disappearance of the effect of quenching, we note an interesting feature in the resistivity curve in Fig. 1c. There is a small but evident anomaly at about 385 K. To examine this anomaly, resistivity measurements were carried out for two transformation cycles with heating ranges below and above 385 K. The result is shown in Fig. 2. As is clearly seen in Fig. 2, the resistivity decreases on transformation to the martensite, by cooling after heating above the temperature which marks the beginning of the anomaly (Fig. 2a), while the resistivity increases on transformation to the martensite during thermal cycling below this temperature. This result indicates that the resistivity anomaly at about 385 K is associated with the process of the disappearance of the quenching effect. Henceforth the temperature at which the anomaly begins is referred to as Q_a.

Fig. 3 shows the resistivity curves of the quenched Au–49.0 at%Cd alloy. The resistivity anomaly is also found at about 371 K; it is considerably larger in comparison with that of the Au–47.5 at%Cd alloy. In addition, Fig. 3a and b indicate that after heating above Q_a, successive cooling and reheating cause a narrow transformation hysteresis, while thermal cyclings below Q_a cause some extension of both the ranges from M_s to M_f, the martensite start and finish temperatures, respectively, and A_s to A_f, the austenite start and finish temperatures. The resistivity behaviour after heating above Q_a in Fig. 3a is similar to that in a slow-cooled case. Therefore, it appears that the resistivity anomaly reflects the mechanism of the disappearance of the quenching effect, as described in the case of the quenched Au–47.5 at%Cd alloy.

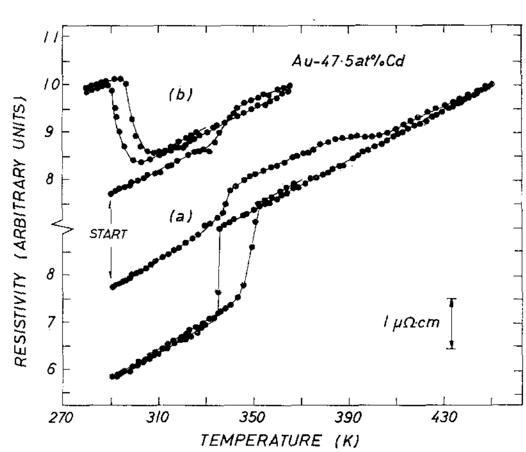

Figure 2 Resistivity curves of the quenched Au–47.5 at%Cd alloy for thermal cycles with heating (a) above 385 K and (b) below 385 K.

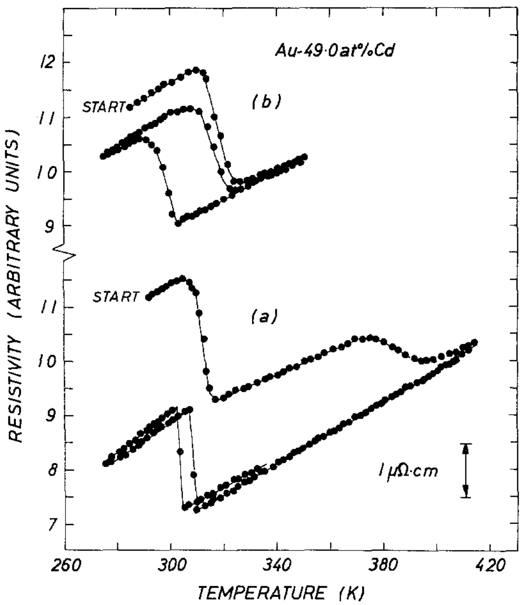

Figure 3 Resistivity curves of the quenched Au–49.0 at%Cd alloy for thermal cycles with heating (a) above 371 K and (b) below 371 K.

In rare cases, the initial transformation temperature is lowered below room temperature by quenching, as shown in Fig. 4. In a previous study [12], the same resistivity behaviour was observed.

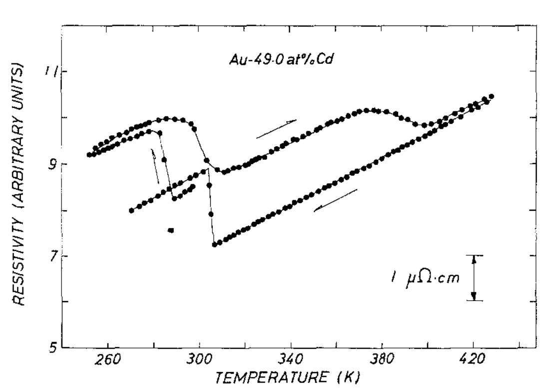

Figure 4 Resistivity against temperature curves for the quenched Au–49.0 at%Cd alloy. Note: the initial transformation is lowered below room temperature.

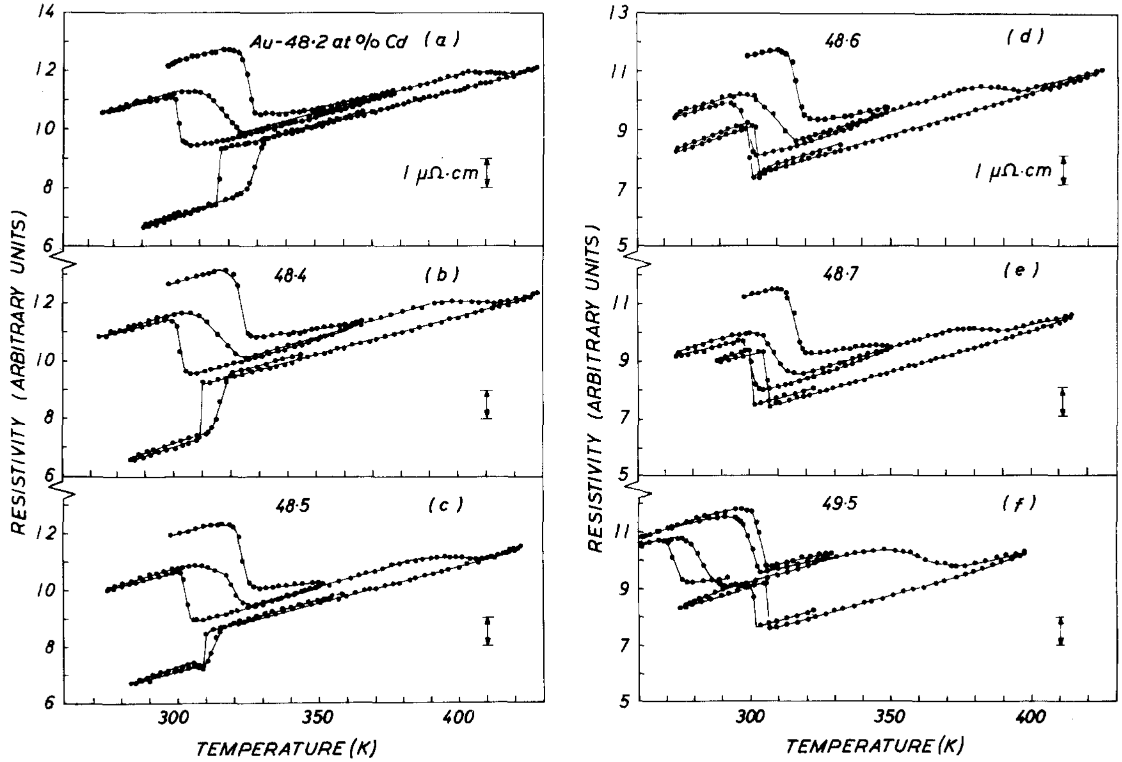

Figure 5 Resistivity against temperature curves for quenched alloys containing 48.2–49.5 at%Cd.

Such a trend becomes marked with increasing Cd content. Actually, in the Au–49.5 at%Cd alloy, quenching always depresses the initial transformation temperature below room temperature (see Fig. 5f).

In order to elucidate the resistivity anomaly, resistivity measurements were extended into the range 47.5 to 49.5 at%Cd. Fig. 5 shows typical examples of the results: details will be reported elsewhere [13]. Q_a and the magnitude of the resistivity drop, defined as $[\rho_h(Q_a)-\rho_c(Q_a)]/\rho_c(Q_a)$, are plotted against Cd contents in Fig. 6. Here $\rho_h(Q_a)$ and $\rho_c(Q_a)$ are the resistivities at Q_a on heating and cooling, respectively. Based on the above results, we can summarize the essential characteristics of the resistivity anomaly as follows: (1) The resistivity drop is more enhanced by increasing Cd contents. In other words, the anomaly is more marked in a composition range in which the ζ_2'-martensite tends to exist as the equilibrium phase; (2) Q_a decreases with increasing Cd content; (3) The anomaly is irreversible, i.e. it appears only on the heating process.

As already stated in previous work [10, 11], it has been considered that quenching has the effect of producing martensites different to those of the equilibrium martensitic phase. In order to clarify this point and to examine the origin of the resistivity anomaly, X-ray diffraction measurements were carried out for the quenched alloys containing 47.5 and 49.0 at%Cd.

Fig. 7 shows parts of the X-ray diffraction patterns of a quenched single crystal of Au–47.5 at%Cd, taken at several temperatures during thermal cycling. Scans were made along the

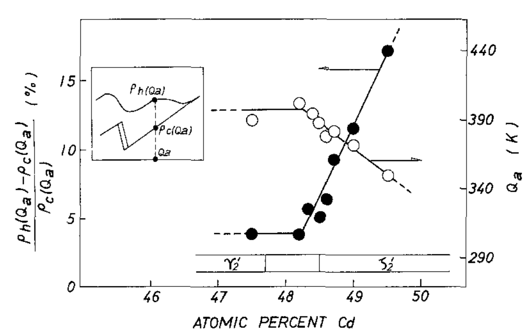

Figure 6 Composition dependence of Q_a and the magnitude of the resistivity anomaly. The magnitude is defined as $[\rho_h(Q_a)-\rho_c(Q_a)]/\rho_c(Q_a)$, where $\rho_h(Q_a)$ and $\rho_c(Q_a)$ are the resistivities at Q_a on heating and cooling, respectively, indicated in the inset. The martensites which exist as equilibrium phases are shown at the bottom, based on the result obtained by Alasafi and Schubert [7].

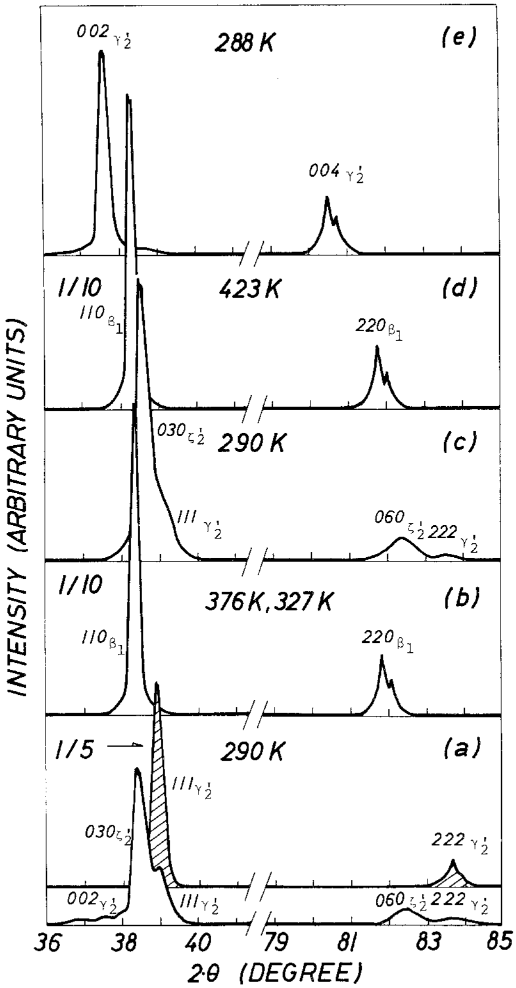

Figure 7 Partial X-ray diffraction patterns of the quenched single crystal of the Au–47.5 at%Cd alloy, measured for two thermal cycles, (a)–(c) and (c)–(e). The scanning direction is parallel to the $\langle 1\,1\,0\rangle_{\beta_1}$ axis. The hatched profile shows the result of measurements taken by rotating the crystal around the $\langle 1\,1\,0\rangle_{\beta_1}$ axis by 1°. The scale of intensity is reduced appropriately as indicated by the fractions.

$\langle 1\,1\,0\rangle_{\beta_1}$ direction. The diffraction peaks shown in Fig. 7a are those obtained from the as-quenched state and they may be considered to be a mixture of the γ_2' and ζ_2'-martensites. That is, the formation of the γ_2' martensite cannot be prevented even when quenched. The results obtained from a Debye–Scherrer photograph also indicated the existence of both γ_2' and ζ_2'-martensites, although the powder method is, in general, inadequate for analysing complicated diffraction patterns from a specimen including several phases. These two kinds of martensites transfrom simultaneously to the β_1-phase on heating to 376 K, and the β_1-phase formed is stable up to at least 327 K on successive cooling cycles (Fig. 7b). On further cooling to room temperature, the γ_2' and ζ_2'-martensites are produced again (Fig. 7c). The above thermal cycle corresponds to that for the resistivity measurement shown in Fig. 1a. From comparison of the above results with the resistivity curves of Fig. 1a, it can be seen that the slight increase in resistivity appearing at about 332 K on the initial heating process is due to the simultaneous $\gamma_2' \rightarrow \beta_1$ and $\zeta_2' \rightarrow \beta_1$ transformations. The hysteresis in the vicinity of 300 K on successive cooling and reheating is also attributed to the $\beta_1 \leftrightarrow \gamma_2'$ and $\beta_1 \leftrightarrow \zeta_2'$ transformations. It would be interesting to know the volume ratio of the γ_2' and ζ_2'-martensites at this stage, but no quantitative information can be obtained, because the diffraction profiles are generated by variants of the γ_2' and ζ_2'-martensites which do not satisfy the diffraction condition along the arranged scanning direction exactly. Indeed, as given by hatched profiles in Fig. 7a, the intensity of the diffraction peaks are changed greatly by tilting the crystal by about 1°.

As mentioned above, this alloy exhibits the resistivity anomaly at about 385 K when quenched. However, Fig. 7b and d show that only the β_1-phase is detected both below and above Q_a. This result indicates that the resistivity anomaly can not be attributed to a direct phase transformation.

In this work, no X-ray diffraction analysis was undertaken for the transformation process corresponding to Fig. 1c. Nevertheless, it is reasonable to consider that the dominant transformation is the $\beta_1 \leftrightarrow \gamma_2'$ one, because in this thermal cycle, the specimen was heated near to Q_a, which leads to a decrease in quenching effect.

Fig. 8 shows part of the X-ray diffraction patterns of the quenched single crystal of the Au–49.0 at%Cd alloy, taken along the $\langle 1\,1\,0\rangle_{\beta_1}$ direction at several temperatures. Clearly, the $\beta_1 \leftrightarrow \zeta_2'$ transformation occurs even though the specimen is quenched but no diffraction peaks which could be attributed to γ_2' were found, even when the crystal was tilted about the $\langle 1\,1\,0\rangle_{\beta_1}$ axis. The presence of a very small amount of the γ_2'-martensite in a powder specimen [12] was probably due to the difference in transformation characteristics of the powder and bulk samples.

Fig. 8b and c show that the large resistivity anomaly at about 371 K occurs in the β_1-phase. This result is the same as that for the quenched Au–47.5 at%Cd alloy.

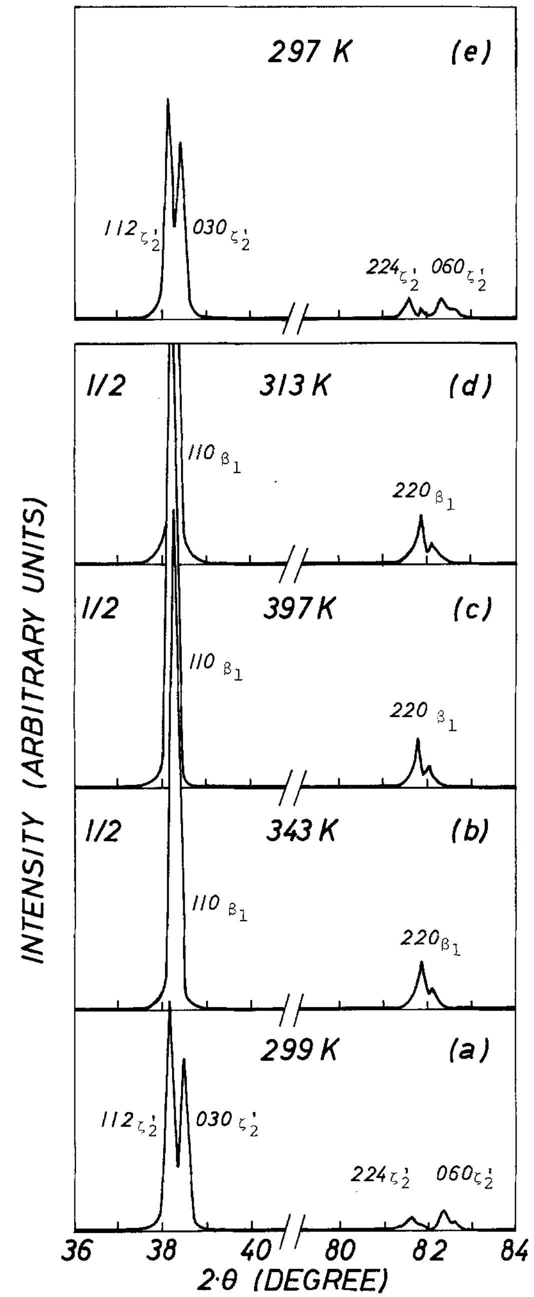

Figure 8 Partial X-ray diffraction patterns of the quenched single crystal of the Au–49.0 at%Cd alloy on heating and cooling, measured along the $\langle 1\,1\,0 \rangle_{\beta_1}$ direction. The scale of intensity is reduced appropriately as indicated by the fractions.

It is well known that alloys which undergo an order–disorder transformation exhibit a large increase in resistivity when quenched from high temperatures. It is therefore possible that the resistivity anomaly is an ordering reaction. Fig. 9 shows the intensity variation with temperature of the 1 1 0 superlattice reflection of the β_1-phase of

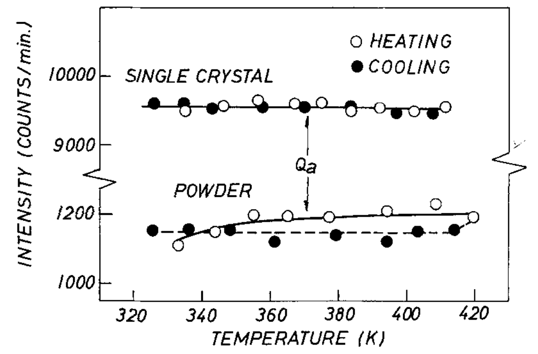

Figure 9 Intensity of the β_1 1 0 0 superlattice reflection as a function of temperature for the quenched Au–49.0 at%Cd alloy on heating and cooling.

the quenched Au–49.0 at%Cd alloy. No detectable change could be found in the vicinity of Q_a. Previously, Wechsler [14] measured the intensity of the 1 0 0 reflection with changing temperature using an annealed powder sample of Au–48.5 at%Cd and obtained a definite superlattice reflection even at 873 K. Hence, the resistivity anomaly can not be explained in terms of the ordering reaction.

Additional quenching experiments were performed from various elevated temperatures for the Au–49.0 at%Cd alloy to obtain the quenching temperature dependence of the magnitude of the resistivity anomaly. Fig. 10 indicates that Arrhenius linearity is satisfied, and suggests that the resistivity anomaly is related to a thermally-activated process, with an activation energy of about 0.35 eV. Details concerning the resistivity anomaly will be discussed again in the following section.

The results obtained in this work are summarized as follows: (1) By quenching from 723 K into ice water, the Au–47.5 at%Cd alloy transforms from the β_1-phase to both γ'_2 and ζ'_2-martensites and the $\beta_1 \leftrightarrow \gamma'_2$ and $\beta_1 \leftrightarrow \zeta'_2$ transformations occur simul-

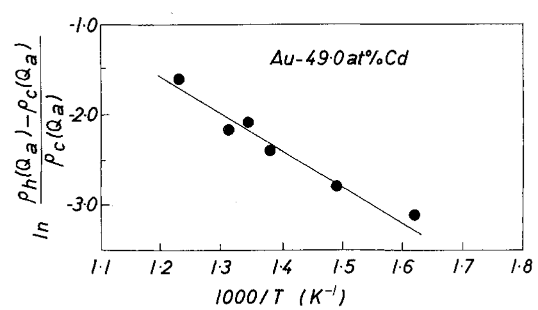

Figure 10 Logarithm of the magnitude of the resistivity anomaly against reciprocal quenching temperature for the Au–49.0 at%Cd alloy. The magnitude of the anomaly is given as $[\rho_h(Q_a) - \rho_c(Q_a)]/\rho_c(Q_a)$.

taneously if the thermal cycles are performed in the temperature range below the temperature of beginning of the resistivity anomaly, Q_a. The resistivity changes and transformation temperatures are different between the first heating run and the following thermal cycles, although in both cases the simultaneous $\beta_1 \leftrightarrow \gamma_2'$ and $\beta_1 \leftrightarrow \zeta_2'$ transformations occur. These simultaneous transformations disappear when heated above Q_a (~385 K); the following thermal cycle results in the usual $\beta_1 \leftrightarrow \gamma_2'$ transformation; (2) the transformation characteristics of the Au–49.0 at%Cd alloy, on the other hand, remain structurally unaffected by quenching, i.e. the $\beta_1 \leftrightarrow \zeta_2'$ transformation predominates. However, quenching leads to an extension of the M_s–M_f ranges. As in the case of the Au–47.5 at%Cd alloy, the effect of quenching disappears when heated above Q_a (~371 K); (3) the resistivity anomaly becomes more pronounced and Q_a decreases with increasing Cd content. The origin of the resistivity anomaly is related to a thermally-activated process, but could not be attributed to the ordering reaction.

4. Discussion

The above results indicate that in the Au–47.5 at%Cd alloy the ζ_2'-martensite is the characteristic product under quenching conditions, but always co-exists with the equilibrium γ_2'-martensite. Since the same quenching effect is expected for the Au–49.0 at%Cd alloy, two types of ζ_2'-martensite would be present in the quenched alloy; one is due to the quenching and the other is a product of the equilibrium phase.

Very recently, Tadaki and Wayman [15] examined the quenching effect in the Au–47.5 at%Cd alloy using optical microscopy, electron microscopy and diffraction. They found that three kinds of martensites, γ_2', ζ_2' and β_2' (modified 9R), were produced on quenching from 773 K into ice-water. The reason for this discrepancy is not clear, but there seems to be some difference in the transformation mechanisms of thin films and bulk specimens.

It is interesting to note that the resistivity behaviours are very complicated when the $\beta_1 \leftrightarrow \gamma_2'$ and $\beta_1 \leftrightarrow \zeta_2'$ transformations occur simultaneously, as shown in Fig. 1. This indicates that the process of simultaneous transformations is extremely sensitive to thermal treatment conditions such as quenching temperature and thermal cycling. Actually, a wide variation in resistivity behaviour with quenching temperature was found in Au–47.5 at%Cd [11].

Next, we will discuss the origin of the resistivity anomaly in more detail. Since many vacancies might be frozen into the lattice by quenching from high temperatures it is possible that the quenched-in vacancies are responsible for the resistivity anomaly. Previous work by Wechsler [14] has shown that for the Au–49.0 at%Cd alloy quenching from 723 to 313 K produced a resistivity increase of the order of 1 $\mu\Omega$cm. This value is consistent with the magnitude of the resistivity anomaly obtained from the Au–49.0 at%Cd alloy shown in Fig. 3a. Therefore, the large excess resistivity upon quenching reported by Wechsler corresponds to the resistivity anomaly found in the present study. On the assumption that the excess resistivity is due to the quenched-in vacancies, Wechsler obtained a value of 0.38 eV for the energy of formation of such vacancies. Furthermore, Sturm and Wechsler [16], using resistivity and density measurements obtained a similar result, with a formation energy of about 0.28 eV. As described above, the activation energy determined by the measurements of the magnitude of the resistivity anomaly with quenching temperature was about 0.35 eV. This value is close to the formation energy mentioned above. Taking into account that no structural change is found near Q_a it is therefore reasonable to consider that the resistivity anomaly may arise from the annihilation of the quenched-in vacancies.

Based on these results, the following transformation mechanism can be proposed for the quenched alloys. The quenched-in vacancies cause some local lattice instabilities and act as a trigger for the $\beta_1 \leftrightarrow \zeta_2'$ transformation. In the case of Au–47.5 at%Cd, therefore, the $\beta_1 \leftrightarrow \zeta_2'$ transformation occurs in addition to the $\beta_1 \leftrightarrow \gamma_2'$ transformation. Such a transformation disappears when the quenched-in vacancies disappear by heating above Q_a. The following thermal cycles result only in the $\beta_1 \leftrightarrow \gamma_2'$ transformation. In the composition range where the γ_2'-martensite tends to exist as the equilibrium phase, the transformation process is considered to be similar to that for the Au–47.5 at%Cd alloy. In the case of the Au–49.0 at%Cd alloy, on the other hand, only the ζ_2'-martensite is produced even when quenched. This behaviour seems quite natural, because quenching has the effect of promoting the $\beta_1 \leftrightarrow \zeta_2'$ transformation. The transformation process in this alloy can thus be proposed as in the case of the Au–47.5 at%Cd alloy. That is, when thermal cycles are performed below Q_a, the excess vacancies introduced by quenching remain stable and thus affect the transformation characteristics of the $\beta_1 \leftrightarrow \zeta'_2$ transformation such as the transformation temperature. However, when the alloy is heated above Q_a, the quenched-in vacancies disappear and subsequent thermal cycles result in the usual $\beta_1 \leftrightarrow \zeta'_2$ transformation with a small, sharp temperature hysteresis.

There is still a question regarding why vacancies act as the trigger for the $\beta_1 \leftrightarrow \zeta'_2$ transformation. Although the answer to this question is not known at present, the theoretical model proposed by Paemel *et al.* [17] may be a guide. According to their calculation, if a clustering reaction of vacancies occurs in β-brass, taking account of the elastic anisotropy, it is possible that local lattice rearrangements take place and then an ω-like structure is formed. Of course, since the nucleation process and the crystal structure are different between the ζ'_2 and ω-phases, the above model cannot be applied directly to the formation of the ζ'_2-martensite. However, it is worthwhile pointing out that both phases appear by quenching and show similar features such as a very small volume change and complicated atom displacements through the transformation from the β-phase.

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