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Electrical Properties of Ta_2H[†]

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Data have been obtained on the electrical resistivity, Hall effect, and thermoelectric power in Ta_2H from low temperatures to above the hydrogen sublattice order-disorder transformation temperatures. Prominent manifestations of the β_1-β_2 and β_2-α transformations are evident in the data, but no clear evidence was found for a splitting of the upper transition such as that reported in the calorimetric studies of Saba *et al.* Several features of the electrical properties are consistent with the existence of similar conduction-electron dispersions in Ta_2H and Ta metal, but other factors appear to be inconsistent with this possibility. Hysteresis was observed in the electrical resistivity in the vicinity of the order-disorder transformations, and possible origins of this hysteresis are discussed. The Hall-effect data show that the majority charge carriers in Ta_2H are holes.

I. INTRODUCTION

Tantalum hydride of nominal composition Ta_2H has been studied extensively both experimentally[1,2] and theoretically.[3] Interest in this interstitial system centers on the order-disorder transformations which take place in the hydrogen sublattice as the temperature is increased. At temperatures below ∼308°K, Ta_2H exists in the β_1 form characterized by a pseudotetragonal Ta sublattice with $a \approx b \approx \sqrt{2}a_0$ and $c \approx a_0$, where a_0 is the lattice parameter of bcc Ta metal. Neutron scattering experiments[4] on Ta_2D have been interpreted[5] as evidence that the hydrogen atoms in $\beta_1 = Ta_2H$ occupy the tetrahedral positions $(\frac{1}{4}\,\frac{1}{4}\,\frac{1}{4})$ and $(\frac{1}{4}\,\frac{3}{4}\,\frac{3}{4})$, so that in this phase long-range order exists in the hydrogen sublattice. This order is partially destroyed in β_2-Ta_2H, which exists between ∼308 and ∼332°K. Evidence from specific-heat measurements by Saba *et al.*[6] suggests that still another form, β_3-Ta_2H, might exist over a small temperature range (∼2°K) above ∼332°K, but electrical-resistance data reported by Wallace[4] show no evidence to support this possibility. Finally, above ∼334°K the interstitial hydrogen is thought to be completely disordered, and the Ta sublattice assumes the bcc or α structure characteristic of Ta metal.

The work reported here was undertaken with the hope that more extensive data on the electrical properties would shed light on the nature of the order-disorder transformations and on the way in which addition of hydrogen to Ta influences the electronic structure. Some of the results presented below are consistent with a high degree of similarity between the conduction-electron structures in Ta_2H and in pure Ta metal, but other factors are in seeming conflict with this interpretation.

II. EXPERIMENTAL METHODS

The Ta_2H used in this investigation was prepared according to standard massive hydriding techniques.[1,2] The final composition, $TaH_{0.502}$, closely approached the desired stoichiometry. X-ray data obtained on this material are listed in Table I. They are in good agreement with other determinations.[7,8]

A single specimen, of dimensions $0.012\times0.1365\times0.875$ in.^{3}, was used for most of the electrical measurements, but, as indicated later, the thermoelectric-power data were augmented with measurements on other specimens cut from the same massively hydrided material.

The techniques used for the electrical-resistivity and Hall-effect measurements have been described elsewhere.[9,10] The thermoelectric-power apparatus used in this study differed from that employed in our earlier work[11] in that provision was made to control the hot and cold junction temperatures separately. This was necessitated by the strong temperature dependence of the thermoelectric power of Ta_2H in the vicinity of the order-disorder transformations.

Table I. X-ray lattice parameters for the various phases of Ta_2H.

Phase	T (°K)	a (Å)	c (Å)	c/a
β_1	293	3.369	3.403	1.0101
β_2	329.5	3.368	3.401	1.0098
α	349	3.383		

III. EXPERIMENTAL RESULTS

A. Electrical Resistivity

The electrical resistivity of Ta_2H is plotted as a function of temperature in the range 4–408°K in Fig. 1. The measurements show reasonable qualitative accord with Ta_2H electrical-resistance data reported earlier by Wallace[4] for the temperature range 300–350°K, but a detailed comparison is not possible because the Wallace data were not reduced to absolute resistivity values. The steps which are completed at 308 and 332°K correspond, respectively, to the β_1-β_2 and β_2-α order-disorder transformations. No pronounced evidence was found for a splitting of the upper transition such as that revealed in the specific-heat and differential-thermal-analysis data of Saba *et al.*[6] However, a very slight bump is just perceptible in our resistivity data between 332 and 338°K.

Also included in Fig. 1 for comparison are data obtained by Rosenberg[12] on the electrical resistivity of pure Ta metal. Rosenberg's data have been shifted upward by 2.375 $\mu\Omega$ cm, so that the ideal resistivities of Ta_2H and Ta may be compared more directly. The ideal resistivity $\rho_i(T)$ is defined here by the expression

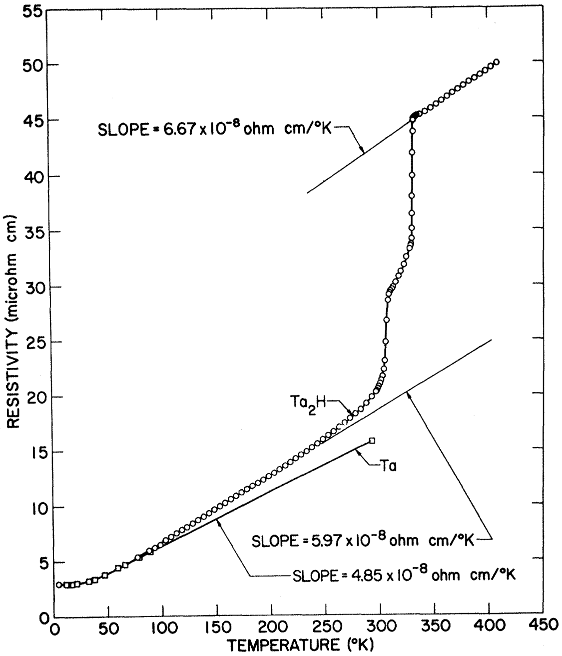

Fig. 1. Electrical resistivity versus temperature for Ta_2H. The data of Rosenberg (Ref. 12) on Ta (shifted upward by 2.375 $\mu\Omega$ cm) are also included for comparison.

$$\rho(T)=\rho_r+\rho_i(T), \tag{1}$$

where $\rho(T)$ is the total resistivity at the temperature T, and ρ_r is the residual resistivity or the normal-state resistivity at $T=0$°K. It is interesting that below the

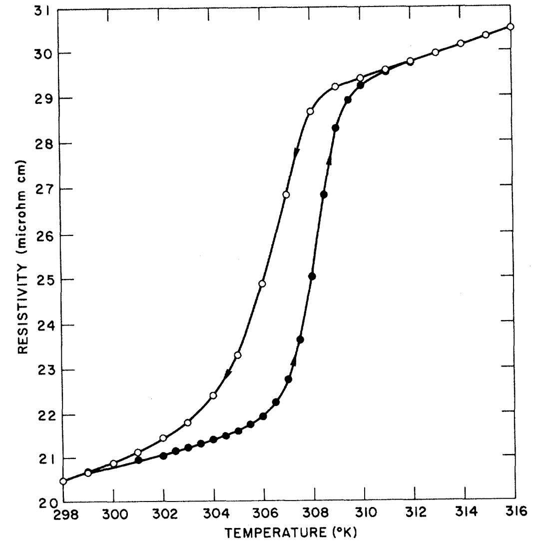

Fig. 2. Electrical resistivity versus temperature for Ta_2H, showing hysteresis in the vicinity of the lower-temperature order-disorder transformation.

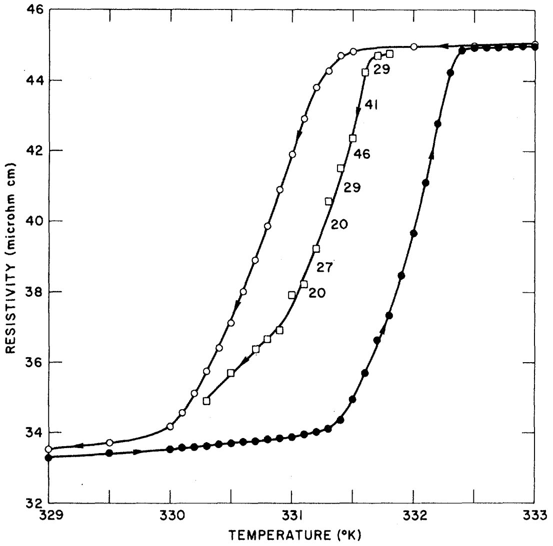

FIG. 3. Electrical resistivity versus temperature for Ta_2H, showing hysteresis in the vicinity of the higher-temperature order-disorder transformation. A closer approach to equilibrium is indicated by the center branch, where the numbers indicate the time interval between successive points in minutes.

ordering temperature of the hydride the ideal resistivities of Ta_2H and Ta differ by a relatively small amount. This will be the subject of further discussion in Sec. IV A.

Hysteresis was observed in the resistivity of Ta_2H in the vicinities of both order-disorder transitions. The scale of Fig. 1 precludes an adequate presentation of the observed effects, and so the results for the β_1-β_2 and β_2-α transitions are presented separately in Figs. 2 and 3. The usual heating and cooling rates were such that 2–5 min elapsed between successive data points. However, an apparent close approach to equilibrium could be achieved by marked reduction of the rate of temperature change. This is illustrated in Fig. 3, where the numbers between successive points indicate the time interval in minutes between these points.

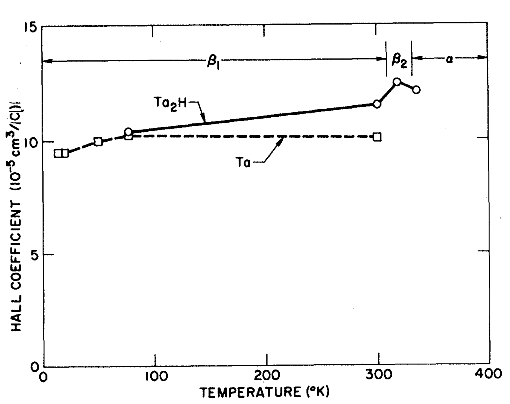

FIG. 4. Hall coefficient versus temperature for Ta_2H. The Ta data of Krautz and Schultz (Ref. 15) are also shown for comparison.

Because of their relevance to the hysteresis results reported above, some unpublished results of Tomasch[13] on the effects of rapid temperature quenching on the electrical resistivity of Ta_2H should be mentioned here. The electrical resistivity of a Ta_2H specimen was measured at 77°K both before and after it was heated to 300°K and then rapidly quenched to 77°K, and also both before and after it was heated to 400°K and then rapidly quenched to 77°K. The resistivities at 77°K were the same to within 0.4% in all cases. This result, together with NMR data[14] which show that hydrogen is not mobile in Ta_2H at 77°K, suggests that the hydrogen-ordering kinetics in Ta_2H are very fast in the vicinity of the order-disorder transitions.

TABLE II. Comparison of properties of β_1-Ta_2H and Ta.

Property	β_1-Ta_2H	Ta
$R_H \times 10^5$ (cm^3/C)	11.52	10.1[a]
$n^* \times 10^{22}$ (cm^{-3})	5.42	6.18
Carriers/Ta atom	1.05	1.12
Θ_D (°K)	250[b]	230[c]
Θ_E (°K)	1680[d]	
$\gamma \times 10^4$ (cal/mole deg^2)	4[e]	14.3±0.5[f]
$\chi \times 10^6$ (emu/g)	0.650[g]	0.844[g]

[a] Reference 15.
[b] Reference 6.
[c] Reference 20.
[d] Reference 23.
[e] Reference 24.
[f] Reference 25.
[g] Reference 7.

B. Hall Effect

The Hall-effect data for Ta_2H were obtained in magnetic fields up to 30 kG, and at all measurement temperatures the Hall voltage was linear in magnetic field strength. The corresponding Hall-coefficient data are presented in Fig. 4 together with the data of Krautz and Schultz[15] for Ta metal. The temperature dependence of the Hall coefficient in Ta_2H is quite small even though the data span the order-disorder transitions. Moreover, differences between the values for Ta_2H and for Ta metal are relatively small. The positive sign is of course indicative of majority hole conduction. Table II compares room-temperature Ta_2H and Ta values for n^*, the "effective" number of charge carriers per cm^3, deduced from the expression

$$n^* = 1/R_H e c, \tag{2}$$

where R_H is the Hall coefficient, e is the electron charge, and c is the speed of light. The parameter n^*

is doubtless a grossly simplistic representation for the actual energy-band structures of Ta_2H and Ta. Nevertheless, it is of interest, because when the n^* values are combined with the Ta atom densities in these two materials, it is found that the effective numbers of charge carriers per Ta atom are nearly the same: 1.05 carriers/Ta atom in Ta_2H and 1.12 carriers/Ta atom in Ta.

C. Thermoelectric Power

The thermoelectric-power data are shown in Fig. 5. Three different specimens were studied, and a number of runs were made as indicated by differently designated points. The data as plotted represent thermoelectric power of Ta_2H against Cu. The dashed line[16,17] is the absolute thermoelectric power of Cu, which *in this temperature range* follows the form $-0.385+0.0069T$ in $\mu V/°K$. The absolute thermoelectric power of Ta_2H would be given by the sum of the dashed line and the experimental points. It is apparent that the absolute thermoelectric power is small and negative in β_1-Ta_2H and is positive in the β_2 and α phases. Only two order-disorder transitions are clearly evident. Thus, even though the thermoelectric power is a highly sensitive indicator of changes in electronic structure and electron scattering,[18] there is no definite indication of a splitting in the upper transition such as that reported in the calorimetric studies.[6] On the other hand, even though the data exhibit considerable scatter, the trends for single runs seem to indicate that a slight bump, perhaps a bit more prominent than that observed in the electrical resistivity, exists just above the upper transition. This bump has been indicated schematically by the solid line drawn in the midst of the scattered data.

No data on the thermoelectric power of pure Ta against Cu in this temperature range are available for comparison.

IV. INTERPRETATION AND DISCUSSION

A. Comparison of Electronic Strucures of Ta_2H and Ta

Several, although not all, factors seem to suggest that those portions of the electronic structure which participate in electron transport (i.e., the conduction-electron dispersions) are not greatly different in Ta_2H and Ta. Other factors seem to be in conflict with this view. Data bearing on this point are listed in Table II. As noted above, the observed Hall coefficients for Ta_2H and Ta differ by only a relatively small amount despite the occurrence of the order-disorder transformations in the hydride. This is particularly surprising in view of the fact that the Hall coefficient is generally a very sensitive indicator of changes in both the electronic structure and scattering mechanisms.[9,10,18,19] Viewed in terms of the number of charge carriers per Ta atom in β_1-Ta_2H and Ta, the difference is even smaller: 1.05 carriers/Ta atom in Ta_2H and 1.12 carriers/Ta atom in Ta.

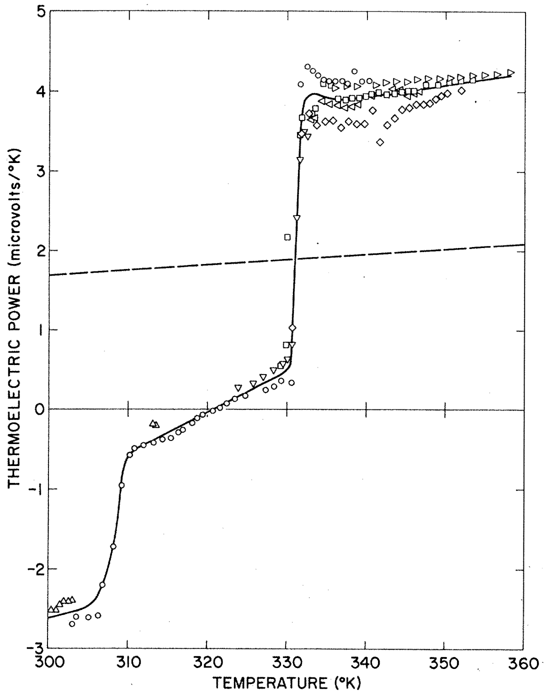

FIG. 5. Thermoelectric power versus temperature for Ta_2H against Cu (data points). The dashed line represents the absolute thermoelectric power of Cu (Refs. 16 and 17).

Several aspects of the electrical-resistivity data can also be explained in terms of similar conduction-electron dispersions in Ta_2H and Ta. We develop this argument by first assuming the conduction-electron dispersions in these two materials to be the same. Then we examine the consequences of differences in the lattice vibration spectra of the two materials, and finally we consider the effects of electron scattering from the disordered (or "melted") hydrogen sublattice in the Ta_2H. In Ta the lattice vibrations are assumed to be Debye-like and hence can be characterized (see Table II) by a Debye temperature $\Theta_D=230°K$ as derived from specific-heat measurements.[20] The corresponding temperature dependence of the electrical resistivity should follow approximately the well-known Grüneisen function[21] for this characteristic temperature.

In Ta_2H, by analogy with zirconium hydride,[22] the phonon dispersion should consist of a low-energy acoustic branch, which is approximately Debye-like, and, in addition, a much-higher-energy optical branch which is Einstein-like, with the hydrogen atoms vibrating much like independent single-frequency simple-harmonic oscillators. Specific-heat measurements[6] on Ta_2H yield the value $\Theta_D=250°K$ for the characteristic temperature of the acoustic branch.

Inelastic neutron scattering data[23] for the composition $TaH_{0.7}$ correspond to an Einstein or optical-branch characteristic temperature of $\Theta_E = 1680°K$. This value is most likely a satisfactory approximation for the composition Ta_2H as well, inasmuch as Θ_E varies only slightly with hydrogen concentration in other hydrides.[23] On the basis of the above, it is now possible to speculate on the ideal resistivities of Ta_2H and Ta. Three factors suggest that the ideal resistivities should be nearly the same at low temperatures. First, nearly identical conduction-electron structure was assumed from the outset. Second, the Debye temperatures in Ta_2H and Ta are nearly the same: 250 versus 230°K. Third, the Einstein temperature $\Theta_E = 1680°K$ is so high that only very few of the Einstein-oscillator states will be excited at low temperatures and hence available to scatter electrons. In fact, by analogy with zirconium hydride,[11] scattering from the Einstein modes is expected to become significant only above about 200–250°K. The upturn evident in the Ta_2H data between 230 and 290°K could well be an indication of the onset of optical-mode scattering. This upturn might also arise in part from the onset of the β_1-β_2 order-disorder transition. Although NMR motional narrowing experiments[14] provide evidence that hydrogen first shows signs of mobility in Ta_2H in the vicinity of $\sim$100°K, the hydrogen motion would not be expected to influence the electrical resistivity appreciably. Not only is the lifetime of the hydrogen atoms in their sites much larger than the jump time,[14] but also the ratio of octahedral to tetrahedral site occupancy by hydrogen atoms is believed to be small at room temperature and below.[14]

When the order-disorder transformations take place, an enormous increment of disorder scattering, $\sim 24\ \mu\Omega$ cm, is added to the electrical resistivity. However, the electronic structure does not appear to be altered appreciably, judging from the fact that the slopes of the resistivity-temperature curves above and below the transition temperatures differ by only $\sim$10%. This suggests, in effect, that the "melting" of the hydrogen sublattice introduces a very large nearly temperature-independent hydrogen disorder scattering while at the same time leaving relatively unchanged the scattering of electrons by acoustical-mode phonons.

Taken together, all of the above factors are consistent with the original hypothesis of similar conduction-electron dispersions in Ta_2H and Ta. On the other hand, in apparent conflict with this possibility are the data[24,25] shown in Table II for the electronic specific-heat coefficient γ, which is a measure of the electronic density of states at the Fermi level. The seriousness of this difference is difficult to assess, because the γ value for Ta_2H appeared only as a footnote in Ref. 6, and no estimate was given of probable limits of error. A difference between the magnetic susceptibilities[7] of Ta_2H and Ta is also indicated in Table II, but this is a less troublesome difference, because sources in addition to the conduction electrons also contribute to the magnetic susceptibility.

The charge state of hydrogen in metallic hydrides has been the subject of extensive discussion,[1] and the present results are relevant to this question. If, as the Hall-effect and resistivity data suggest, the conduction bands in Ta_2H and Ta are nearly the same, this would imply that hydrogen exists in its neutral atomic form in Ta_2H. This conclusion would be without basis, of course, if the electron-transport similarities were merely a fortuitous combination of diverse factors, as suggested by the electronic specific-heat discrepancy. Additional data on the electronic specific heat of Ta_2H would be of value in resolving this question.

B. Residual Resistivity

The value $\rho_r = 3.00\ \mu\Omega$ cm obtained for the residual resistivity of Ta_2H in this study requires some comment. If the only defects were those arising from lack of stoichiometry of the $TaH_{0.502}$ specimen, then a much smaller value of ρ_r would be expected. This conclusion is based on the result that if complete disordering of the hydrogen sublattice in the disordering transformations adds $\sim 24\ \mu\Omega$ cm, then the defect concentration $c = 0.004$ in $TaH_{0.502}$ should yield $\rho_r = 0.38\ \mu\Omega$ cm, assuming proportionality of ρ_r to $c(1-c)$ is appropriate. There are several possible explanations for this discrepancy. The first and most likely one is that grain boundaries, dislocations, and microcracks are responsible. Second, if the hydrogen analysis were in error by 2.8%, and the defect concentration were 0.032 instead of 0.004 (which seems unlikely), then the departure from stoichiometry alone could account for the observed ρ_r. Third, a few atomic percent of substitutional and/or interstitial impurities in the Ta used to form the hydride could produce the observed ρ_r, but the nominal impurity level in the Ta used to form the hydride was $<0.1\%$. Most likely all three of the above factors contribute in part to the observed ρ_r. Another factor which can probably be ruled out on the basis of the Tomasch quenching experiments is the possibility of quenched-in hydrogen defects. Finally, the observed ρ_r might be ascribed in part to the intrinsic disorder first proposed by Wallace[4] to explain his neutron-diffraction data, but subsequent structure determinations[5] argue against this possibility. Unfortunately the present results fail to provide an answer to the question of whether a pure stoichiometric crystal of Ta_2H would indeed exhibit a null residual resistivity which would be indicative of perfect order.

C. Hysteresis in Order-Disorder Transformations

As shown in Figs. 2 and 3, hysteresis effects were observed resistively in the order-disorder transformations in this investigation. Hysteretic internal friction peaks associated with these transformations have also been reported.[26] In the latter instance the observed hysteresis was attributed to the combination of the supposed slow kinetics and experimental conditions in which the temperature was continuously changed. This same explanation might be invoked to account for the hysteresis reported here for the resistivity, especially in view of the long-time-interval data shown in Fig. 3. On the other hand, several factors appear to be inconsistent with this explanation. First, the NMR data[14] show that hydrogen is highly mobile in Ta_2H at the order-disorder transformation temperatures. Second, the results of the quenching experiments[13] mentioned earlier imply very fast kinetics. A third and final factor is to be found in Figs. 2 and 3, where it is evident that in three out of four cases the increasing-temperature and decreasing-temperature curves merge quite exactly just above and just below the transitions. Taken together, all of these factors might be interpreted as evidence that the observed hysteresis has a basis other than simply slow-ordering kinetics. The possibility that one or both of the transformations might be first order (with the observed hysteresis simply being evidence of superheating and supercooling) should be considered. However, Saba *et al.*[6] have interpreted their specific-heat studies of Ta_2H as indicating that the transitions are second-order or λ-type transitions. Accordingly, full understanding of the origin of the observed hysteresis effects will require further investigation.

D. Splitting of Upper Transition

The data obtained in this work show at most only the slightest hint of a splitting in the upper order-disorder transition. The evidence (if it is evidence at all) is so slight that it would seem to be inconsistent with the rather large effects observed in the calorimetric investigations of Saba *et al.*[6] Resolution of this apparent inconsistency must also await further investigation.

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