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Elastic properties of intermetallic AB_2 and AB_5 hydrogen-absorbing compounds

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Abstract

The elastic properties of AB_2 and AB_5 hydrogen-absorbing intermetallic compounds are reviewed. The relevance of these properties to hydrogen absorption is discussed briefly. In many cases the temperature dependence of the elastic constants of the AB_2 compounds is anomalous in that the moduli do not decrease monotonically with increasing temperature. This unusual behavior appears to be associated with electronic structure effects in a number of cases. The absorption of hydrogen by polycrystalline TaV_2 has a profound effect on the aggregate shear modulus, changing both the magnitude and temperature dependence significantly. This effect of hydrogen on the mechanical properties appears to occur via effects on the electronic structure. Few measurements of elastic properties have been reported for hydrogen-absorbing AB_5 intermetallic compounds. The $LaAl_xNi_{5-x}$ system has been investigated by means of measurements on polycrystals prepared by hot isostatic pressing. The moduli decrease about 8% as x increases from 0 to 1.

Keywords: Hydrogen-storage materials; Intermetallics; Elasticity; Ultrasonics

1. Introduction

The elastic constants of solids are important for many reasons. From a practical viewpoint, they describe the linear response of a material to an applied stress. Among the basic mechanical properties that can be obtained from the elastic constants are the bulk modulus, Young's modulus, the shear modulus, and Poisson's ratio. From a fundamental viewpoint, the elastic constants are the second derivatives of a thermodynamic potential with respect to strain. Ultrasonic experiments give the adiabatic elastic constants, in which case the appropriate thermodynamic potential is the internal energy [1]. Thus, the elastic constants are directly related to atomic bonding and structure. They are also related to thermal properties through the Debye theory. At temperatures above absolute zero there is a vibrational contribution to the free energy that is related to the phonon frequencies and hence to the elastic constants.

Elastic constants are of added importance for hydrogen-absorbing materials. There is an attractive elastic contribution to the hydrogen–hydrogen interaction that depends on the bulk modulus [2]. In addition, the elastic energy associated with the precipitation of hydrides depends on the elastic constants [3–5]. The lattice constants of the hydride are usually different than those of the host metal resulting in misfit strains. These strains result in an elastic energy that affects the formation and decomposition of hydrides [6–10]. Section 2 presents a brief review of the elastic constants of solids and discusses their measurement. Section 3 gives a review of the elastic properties of hydrogen-absorbing AB_2 and AB_5 intermetallic compounds. Section 4 provides a brief summary.

2. Elastic moduli of solids and their measurement

The linear elastic response of a solid is described by a generalized Hooke's Law. In Cartesian coordinates the relation between stress σ_{ij} and strain e_{kl} is

$$\sigma_{ij} = \sum_{kl} C_{ijkl} e_{kl} \tag{1}$$

where σ_{ij} is the ith component of a force acting on a unit area with normal along the jth axis, C_{ijkl} are the elastic constants, and e_{kl} is

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$$e_{kl} = \frac{1}{2}\left[\frac{\partial u_k}{\partial x_l} + \frac{\partial u_l}{\partial x_k}\right] \tag{2}$$

where $u_l(x_k)$, $l, k = 1, 2, 3$, is the displacement of a point whose coordinates were x_k before the elastic deformation. It would appear that there are $3^4 = 81$ elastic constants, but it can be shown quite generally [11] that, if no body torques act on the material, the number of independent elastic constants is reduced to 21. Further reductions depend on symmetry. As a result of the simplifications involved in reducing the number of independent elastic constants from 81 to 21, pairs of subscripts can be replaced by just one following the convention

$$11 \leftrightarrow 1,\ 22 \leftrightarrow 2,\ 33 \leftrightarrow 3,\ 23 = 32 \leftrightarrow 4,\ 31 = 13 \leftrightarrow 5,$$
$$12 = 21 \leftrightarrow 6.$$

Hooke's Law becomes

$$\sigma_i = \sum_{j=1}^{6} C_{ij} e_j \tag{3}$$

with $C_{ij} = C_{ji}$. Crystals with triclinic symmetry require 21 independent elastic constants. Higher symmetries require fewer, e.g. orthorhombic requires 9, hexagonal requires 5, and cubic requires 3. It is often the case that one works with polycrystals. If the grains are small compared to the sample size and randomly oriented, then the sample is elastically isotropic with two independent elastic constants which are usually taken to be C_{11} and $C_{44} = \frac{1}{2}(C_{11} - C_{12})$. If single-crystal elastic constant data are available, the polycrystalline moduli may be estimated from the single-crystal values using various averaging methods [12]. Alternatively, the polycrystalline moduli are sometimes measured directly as will be discussed below. Polycrystals with some preferred orientation of the crystallites are described as having texture; more than two independent moduli are required to describe this situation [13], the number depending on the macroscopic symmetry of the specimen. For the isotropic case the bulk (B), shear (G), and Young's (E) moduli, as well as Poisson's ratio (ν), may be calculated from the polycrystalline moduli C_{11} and C_{44} by means of

$$\begin{aligned} B &= \frac{3C_{11} - 4C_{44}}{3}, & G &= C_{44}, \\ E &= C_{44}\,\frac{3C_{11} - 4C_{44}}{C_{11} - C_{44}}, & \nu &= \frac{C_{11} - 2C_{44}}{2(C_{11} - C_{44})}. \end{aligned} \tag{4}$$

The temperature (T) dependence of the elastic constants is directly related to the anharmonic nature of lattice vibrations. The lattice vibrations typically result in a T^4 term at low temperatures and a term linear in temperature at higher temperatures [14]. In metals there is also a contribution from the conduction electrons. For simple metals the electrons contribute a T^2 term at low temperatures [15]. Volume effects, through the thermal expansion of the crystalline lattice, also play a significant role. The overall result, for simple metals, is that the elastic moduli approach 0 K with zero slope and decrease monotonically with increasing temperature. This simple picture is not applicable to materials undergoing phase transitions [16,17], or to materials with more complicated electronic structures [18]. As will be discussed below, the temperature dependence of the elastic moduli of many intermetallic compounds does not follow the simple picture just discussed.

Dynamical methods offer the most accurate means of experimentally determining the elastic moduli of solids. The ultrasonic pulse-echo technique is widely used [19]. It involves the propagation of short pulses of ultrasound between opposite, parallel faces of a specimen. Given the requirements for plane wave propagation, sample dimensions of the order of a cm are needed for the usual measurement frequencies. For single crystals, measurements along various crystalline directions are usually needed which may necessitate preparing multiple specimens. A technique which has received much attention recently is that of resonant ultrasound spectroscopy (RUS) [20–22]. With this method, the vibrational eigenmodes of samples of simple shapes (parallelepipeds, cylinders, spheres) are measured. The elastic moduli are determined from the measured resonant frequencies. Sample dimensions can be of the order of a mm, and all moduli can be obtained from measurements on one specimen. The use of relatively small samples is important for many materials, including metal–hydrogen systems.

3. AB_2 intermetallic compounds

AB_2 type intermetallic compounds have been of interest due to their possible use in high-temperature structural applications [23]. More recently, they have received attention as possible electrode materials in metal hydride batteries [24]. Some of these materials also have interesting electronic and structural instability properties. The brief discussion following, while not exhaustive, will focus on those compounds known to absorb hydrogen [25]. There have been a number of theoretical [26–28] and experimental [29–39] investigations of these compounds.

As discussed in Section 2, the normal temperature dependence of elastic moduli is considered to be a decrease in magnitude with an increase in temperature. This behavior was found for the single-crystal elastic constants of the cubic Laves phases $ZrCo_2$, $HfCo_2$, $CaAl_2$, YAl_2, $LaAl_2$, and $GdAl_2$ which were measured over the temperature range of 4.2 to 300 K [30,31]. The bulk moduli of these intermetallic phases were found to be reasonably well estimated as the arithmetic mean of the bulk moduli of the constituent elements. The materials were broadly placed into different groups according to the number of valence electrons per formula unit. $GdAl_2$

showed [31] magnetic effects on the elastic constants at low temperatures as did [40] polycrystalline $SmFe_2$. The five hexagonal elastic constants of C14 $CaMg_2$ were measured [29] over the temperature range 100–300 K and found to have a normal temperature dependence and a low elastic anisotropy. In contrast to the normal behavior just described, an anomalous temperature dependence was found for the single crystal elastic constants of the rare earth intermetallic compounds [37,38] $CeRu_2$, as well as those [39] of $CeCo_2$ and $LaRh_2$ [41]. The shear moduli, especially $\frac{1}{2}(C_{11} - C_{12})$ in $CeRu_2$, showed anomalous softening as the temperature was decreased.

In a comprehensive study, the elastic constants of single crystal alloys of b.c.c. Nb_xZr_{100-x}, with x ranging from 100 to 30, were measured from approximately 0 K to the melting point (about 2000 K) [42,43]. The sign of the temperature dependence of the single crystal elastic constant C_{44} depended on the value of x which is related to the average number of electrons per formula unit.

The C15 Laves phase compounds formed from elements of columns four and five of the periodic table have received special attention. The polycrystalline shear moduli [44–47] of the Laves phase compounds HfV_2 and ZrV_2 have an unusual temperature dependence with a V-shaped minimum at about 100 K and an anomalous stiffening with increasing temperature above 100 K up to the vicinity of the melting temperature (~1500 K). An experiment [48] on a single crystal of HfV_2 showed that the shear constant $\frac{1}{2}(C_{11} - C_{12})$ softens by about 45% as the temperature decreases from room temperature to 118 K and then stiffens as the temperature is lowered further to liquid helium temperatures. A very recent structural study [49] shows that the fcc room temperature C15 structure of this material transforms to a b.c.c. tetragonal phase at 112 K and further transforms to a b.c.c. orthorhombic phase at 102 K.

From the brief survey of AB_2 intermetallic compounds just presented, it is seen that, for many of these compounds, the temperature dependence of the moduli is not what is usually considered normal. In many cases the moduli soften as the temperature is lowered, rather than harden. This behaviour appears to be associated with the more complicated electronic structure found in many of these compounds. The addition of hydrogen to metals changes the electronic structure, in particular the position of the Fermi level is typically raised. Hence, such systems offer the possibility to study the effects of hydrogen on elastic behavior that occur via electronic structure effects. A relatively simple electronic structure model [50–52] has been proposed to explain the unusual elastic behavior of HfV_2, ZrV_2, and TaV_2. We now describe modulus measurements on TaV_2 and illustrate the use of the simple model of electronic structure to explain the key features, including the effects of hydrogen.

Fig. 1 presents a comparison of the shear moduli of TaV_2 and $ZrCr_2$. These two materials have the same C15 structure, but the shear moduli are clearly very different, especially with regard to the temperature dependence. $ZrCr_2$ has one more valence electron than TaV_2. The addition of hydrogen contributes electrons at the Fermi level. Based partly on this consideration, we have carried out a systematic study [53] of the elastic constants of polycrystalline TaV_2H_x. Fig. 2a shows the shear modulus of TaV_2H_x as a function of temperature and hydrogen concentration. The results for TaV_2 are unusual. The modulus approaches zero temperature with zero slope, but increases with increasing temperature up to the highest temperature studied, 345 K. The results for $TaV_2H_{0.06}$ are similar, but with a somewhat weaker temperature dependence. The inset in Fig. 2a for $TaV_2H_{0.10}$ shows a shallow minimum near 40 K. For higher x values, the overall temperature dependence is reversed as compared to TaV_2. The results in Fig. 2a at temperatures above about 225 K and for $x \geq 0.18$ have been corrected for a dispersion effect associated with the hopping of hydrogen among the interstitial sites [53], and thus represent the unrelaxed modulus. For $x \leq 0.10$ the effect was small and this correction was not made.

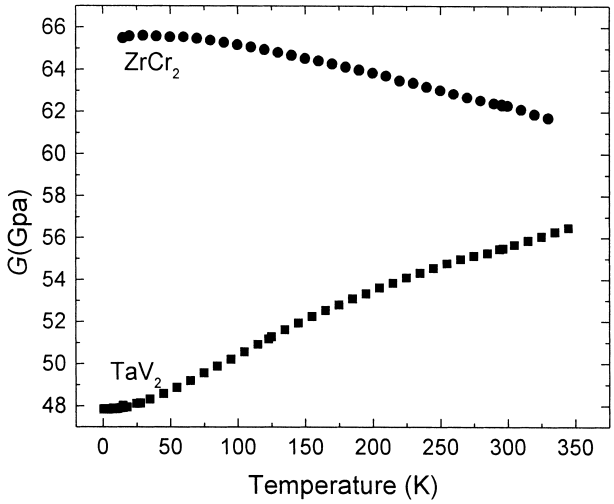

Fig. 1. Elastic shear moduli of polycrystalline $ZrCr_2$ and TaV_2.

The calculation of the electronic contribution to the elastic constants is now outlined. The Helmholtz free energy of a system of N non-interacting electrons each of energy ξ_{bk} is given by [54]

$$F = N\xi_F - 2 \sum_{bk} k_B T \ln \left[1 + \exp\left(\frac{\xi_F - \xi_{bk}}{k_B T} \right) \right] \tag{5}$$

where ξ_F is the Fermi energy, the sum is over all energy bands b and all k values, and the factor 2 accounts for spin. Using $C_{ij}^e = \partial^2 F / \partial e_i \, \partial e_j$ and taking into account that N is conserved gives

$$C_{ij}^e = 2 \sum_{bk} \left[\frac{\partial f}{\partial e_i} \frac{\partial \xi_k}{\partial e_j} + f \frac{\partial^2 \xi_k}{\partial e_i \, \partial e_j} \right] \tag{6}$$

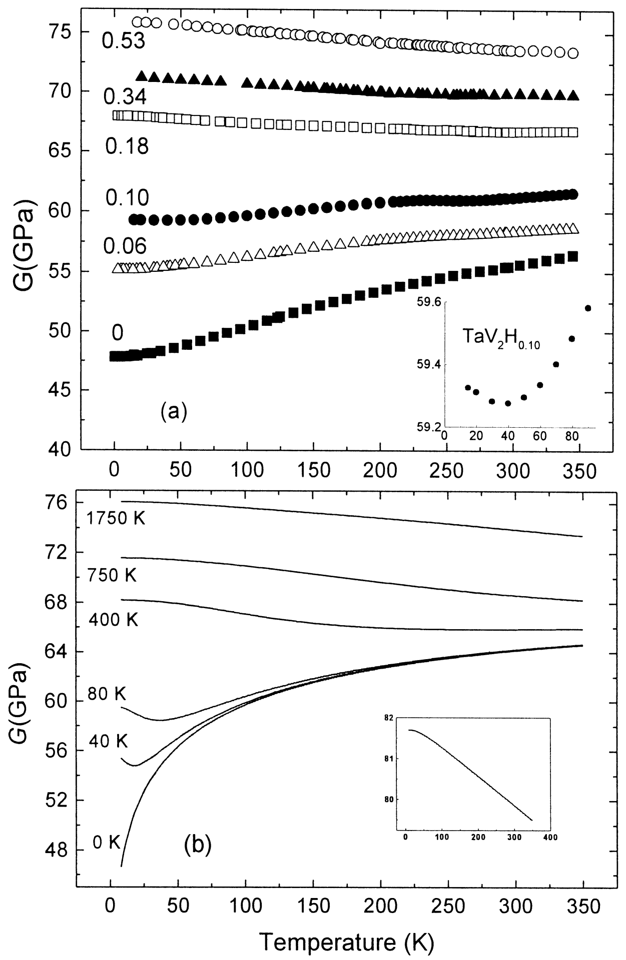

Fig. 2. (a) Elastic shear moduli of polycrystalline TaV$_2$H$_x$ for various x values ranging from 0 to 0.53. These values are listed in the figure. The inset shows the modulus for TaV$_2$H$_{0.10}$. (b) Elastic shear moduli calculated from simple model discussed in the text for various values of the Fermi energy ranging from 0 to 1750 K. These values are relative to the double degeneracy level of the model. The inset shows the background elastic modulus incorporated into the calculations. The background used was the same for all x values.

where f is the Fermi–Dirac distribution function. To proceed further requires specific knowledge of the energy bands and their dependence on strain. Using symmetry arguments, Izyumov et al. [50] proposed a contribution to the electronic structure for C15 materials which seems to be important in some cases, especially TaV$_2$ and possibly HfV$_2$ and ZrV$_2$. The C15 symmetry allows for doubly-degenerate electronic levels at the X-point of the Brillouin zone with a linear dispersion relation in the vicinity of this point. From symmetry considerations these levels couple to a strain e_4. The expression for these levels in the vicinity of the X point is

$$\xi_{1,2}(k) = \pm\sqrt{(sk)^2 + (\gamma e_4)^2} \tag{7}$$

where ξ_F, $\xi_{1,2}$, and k, the electron wave number, are measured with respect to the doubly-degenerate point, s is a proportionality constant, and γ is the deformation potential. Combining Eqs. (6) and (7) gives the result

$$C^e_{44} = \pm 2\gamma^2 \sum_k \frac{f(k_z)}{sk_z}. \tag{8}$$

Eq. (8) offers a simple interpretation of the anomalous temperature dependence of the polycrystalline shear moduli. The lower (upper) branch gives a negative (positive) contribution to the elastic constants. Assume that the Fermi level passes through the double-degeneracy level. (As will be shown, the data indicate that this seems to be approximately the case for TaV$_2$.) At $T=0$ only the lower level is occupied and its contribution to the total shear modulus is negative. As the temperature is raised electrons are excited to the upper branch with its positive contribution. The overall result is that the contribution to the shear modulus due to these bands becomes less negative with increasing temperature resulting in an overall modulus increase with an increase in temperature.

For a more detailed analysis, we set $\varepsilon = sk_z$, express the exponentials in terms of hyperbolic functions, and convert the sum to an integral giving

$$C^e_{44} \propto -\gamma^2 \int_0^\Omega \frac{\mathrm{d}\varepsilon}{\varepsilon}\left[\frac{\sinh(\varepsilon/k_BT)}{\cosh(\varepsilon/k_BT) + \cosh(\xi_F/k_BT)}\right] \tag{9}$$

Eq. (9) can now be used to understand qualitatively the effects of hydrogen. The addition of hydrogen raises the Fermi level in general. Examination of Eq. (9) reveals that C^e_{44} shows a minimum at a temperature $\xi_F/2$. Thus, the minimum in the shear modulus at 40 K for TaV$_2$H$_{0.10}$ implies a Fermi level about 80 K (in temperature units) above the double degeneracy level. We calculated [53] the shear modulus $G(T)$ using

$$G(T,x) = C_{bg}(T) - K \int_0^\Omega \frac{\mathrm{d}\varepsilon}{\varepsilon}\left[\frac{\sinh(\varepsilon/k_BT)}{\cosh(\varepsilon/k_BT) + \cosh(\xi_F/k_BT)}\right] \tag{10}$$

where $C_{bg}(T)$ is a background term representing all the other contributions to the elastic constants such as the ion-core, phonon and other electronic contributions. The constant K is proportional to γ^2, but also includes other factors which are difficult to quantify. Fig. 2b shows the calculations of the shear modulus from Eq. (10) for various values of ξ_F that are listed in Fig. 2b. The background used is shown in the inset. It can be seen that this simple model accounts for the major features of the data; however a caveat is in order. The original model proposed was for a doubly degenerate level at the X point, which would result in effects on the single crystal elastic constant C_{44}. Our measurements are on polycrystals, so it is impossible to determine if effects observed for the polycrystalline shear modulus are due to C_{44}, or perhaps to $\frac{1}{2}(C_{11} - C_{12})$. Also, our results indicate that the Fermi level for TaV_2 passes very near the double degeneracy level, but calculations [52] indicate that the Fermi level is about 6 mRyd (1000 K) above this point in TaV_2. Thus, it might be that the simple model presented above applies, except that some point other than the *X* point may be involved and the single crystal modulus strongly affected might not be C_{44}. Experiments on single crystals are needed to resolve this point.

4. AB_5 intermetallic compounds

There appear to have been relatively few studies of the elastic properties of hydrogen-absorbing AB_5 type intermetallic compounds. The complete set of the elastic constants for hexagonal $SmCo_5$ were measured [55] at room temperature. The complete set of elastic constants of hexagonal $CeNi_5$ were measured [56] over the temperature range 4–300 K. No anomalous behavior was observed. The moduli decreased uniformly with increasing temperature. Three of the five elastic constants were measured [57,58], for hexagonal $PrNi_5$. A strong softening of C_{44} was found as the temperature was lowered below 40 K. This effect was attributed to a strain-quadrupole coupling involving localized 4f electrons.

Turning to the technologically significant material $LaNi_5$, two studies have been reported. Tanaka et al. [59] measured the five single crystal elastic constants of hexagonal $LaNi_5$ at room temperature. Very recently [60], the elastic constants of polycrystalline $LaAl_xNi_{5-x}$ were measured for $0 \leq x \leq 1$. A hot isostatic pressing technique was used to prepare bulk polycrystalline samples of this material that were suitable for ultrasonic measurements. Fig. 3 summarizes the polycrystalline results for the bulk, shear, and Young's modulus. The porosity was calculated by using the difference between the theoretical density (calculated from the measured X-ray lattice parameters) and the measured bulk density. For the *x* values investigated, the porosity ranged from 0 to 2%. Porosity has an effect on the measured moduli [61]. Shown in Fig. 3 are both the measured polycrystalline moduli, and those corrected for porosity. Also shown in Fig. 3 are the polycrystalline average elastic constants calculated [12] from the single crystal values [59]. There are several methods for estimating the moduli of polycrystals from the single-crystal values. The best-known methods are probably the Voigt average, which gives an upper bound, and the Reuss average, which gives a lower bound [12]. The mean of the Reuss and Voigt averages are shown as stars in Fig. 3 for $x = 0$. The calculated bounds for *B*, *G*, and *E* were found to be (127.0, 126.6), (54.8, 56.2), (146.9, 143.70), respectively in units of GPa. The differences between the calculated average, and directly measured, moduli at $x = 0$, while not large, appear to be outside the experimental error. We have no explanation for this difference at this time. The moduli decrease with the substitution of aluminum for nickel, but the effect is not large.

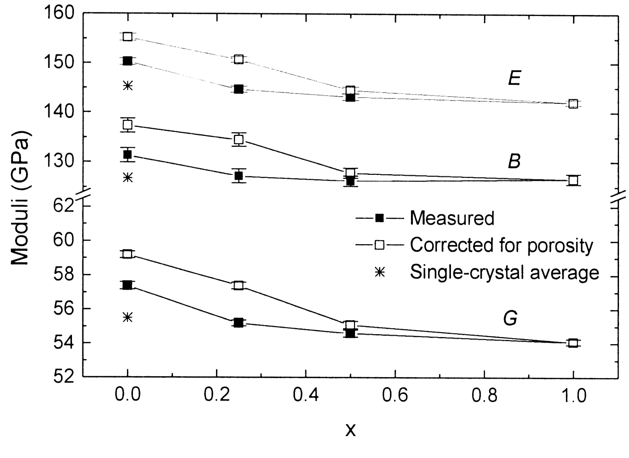

Fig. 3. Polycrystalline Young's (*E*), bulk (*B*), and shear (*G*) moduli for $LaAl_xNi_{5-x}$ as a function of aluminum content, *x*. Both the directly measured, and the porosity-corrected, moduli are shown. The porosity ranged from 0 to 2%.

The moduli of the hydride are often of interest, although it will probably prove difficult to measure them directly. Debye temperatures obtained from heat capacity measurements can be used to estimate the hydride moduli from the hydrogen free results. The long wavelength acoustic contribution to the heat capacity at low temperatures (*T*) is given by

$$c_v = \frac{12\pi^2}{5}\left(\frac{N}{V}\right)k_B\left(\frac{T}{\theta_D}\right)^3 \tag{11}$$

where N/V is usually taken as the number of atoms per unit volume, and

$$\theta_D = \left(\frac{6\pi^2 N}{V}\right)^{1/3}\frac{\hbar}{k_B}v_o \tag{12}$$

with θ_D being the Debye temperature and v_o an average sound velocity. An average sound velocity for the hydrogen-free material can be calculated from the ultrasonic measurements on the polycrystals [60]. An average velocity for the hydrides, v_H, can be determined from heat capacity measurements [62]. Sound velocities are related to elastic moduli by $C = \rho v^2$ where ρ is the density. As a rough approximation we take $C_H = (\rho_H/\rho_o)(v_H/v_o)^2 C_o = fC_o$ where the subscripts H and o refer to the hydride and hydrogen free materials, respectively. For the $LaAl_xNi_{5-x}H_y$ system the factor *f* ranges from 0.46 for $LaNi_5H_{6.78}$ to 0.80 for $LaAl_1Ni_4H_{4.1}$. The dependences on *x* and on *y* are approximately linear between these two limits, so it is difficult to say whether *x* or *y* is more important. In any case, these estimates indicate that the hydride is considerably softer than the hydrogen free material. These moduli are needed to calculate the elastic energy associated with hydride precipitates [60].

5. Summary

Aside from their usual importance, elastic properties have special significance for metal hydrogen materials. Absorption of hydrogen produces an average lattice expansion. This expansion can be related, using the bulk modulus, to an equivalent negative pressure. The result is a negative contribution to the enthalpy of solution that depends on the hydrogen concentration and may be regarded as an effective attractive interaction between dissolved H atoms. In addition, the precipitation of hydrides involves an elastic energy. The details of the elastic energy depend on whether the precipitate is coherent or incoherent, but in any case knowledge of the elastic constants is needed to evaluate this effect. The temperature dependence of elastic constants may be strongly influenced by structural phase transitions. In some cases an anomalous temperature dependence of the elastic constants results from the electronic structure. The elastic constants of a number of hydrogen-absorbing AB_2 intermetallic compounds have been measured. In many cases an anomalous temperature dependence was found that appears to be related to electronic structure. In the case of TaV_2, the absorption of hydrogen has a strong effect on the elastic properties. The mechanism for this effect appears to be via the influence of hydrogen on the electronic structure. This effect suggests that hydrogen may have a strong influence on the elastic properties of other compounds that are especially sensitive to electronic structure. There have been very few measurements of the elastic properties of the hydrogen-absorbing AB_5 compounds. The recent successful application of hot isostatic pressing to the production of samples suitable for ultrasonic measurements may open the way to such measurements on other relevant compounds.

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