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NEUTRON SCATTERING STUDIES OF PREMARTENSITIC PHENOMENA**S. M. Shapiro****Brookhaven National Laboratory
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ABSTRACT

Elastic neutron diffraction and inelastic neutron scattering are ideal techniques for studying premartensitic behavior in metallic alloys. By necessity, real, bulk samples are probed replete with their intrinsic defects. Also, because of the properties of the neutron it is straightforward to probe the behavior of the phonon modes away from the zone center which is probed in the normal ultrasonic techniques. A wide variety of alloys exhibiting martensitic transformations have been studied. It will be shown that most systems undergoing diffusionless transformations exhibit premartensitic behavior in that precursor effects are seen at temperatures well above the martensitic transformation temperature, T_M . This behavior manifests itself in an anomalous temperature dependence of the energy of a particular phonon mode as the temperature approaches T_M . The wavevector of this mode is frequently away from the zone center (i. e., $q \neq 0$). This softening is nearly always accompanied by elastic diffuse scattering at the same wavevector. Particular examples to be discussed are the alkali metals, ω -phase materials and Ni-based alloys

1. INTRODUCTION

The soft mode theory of structural phase transitions is nearly 30 years old. The theoretical idea generated by Cochran and Anderson [1] is quite simple. There is a lattice dynamical mode of vibration whose energy $\hbar\omega$ tends to zero as the structural phase transition temperature is approached. This "soft mode" is clearly a pretransitional effect and the atomic displacements associated with the particular mode determine the low temperature structure. As the frequency becomes zero, the system becomes unstable and it transforms to the low temperature structure. This idea applies to structural phase transitions that are displacive and second order or nearly so. They are also characterized as being diffusionless and continuous. There has been much experimental work on a wide variety of systems that have

confirmed the soft mode theory[2]. The Landau free energy expansion has worked well to describe nearly all these types of transitions. Where needed, it has been modified to include such novel features as incommensurate behavior, coupled order parameters and multiple transitions.

There has been less attention, however, paid to phase transformations that are strongly first order. These systems show an abrupt change in many properties at the transformation temperature. There is usually a large hysteresis and nucleation plays an important role in the kinetics of the phase transitions. In these systems there is frequently a small, sometimes zero, phonon softening and the relationship between the atomic displacements of the anomalous phonon and the low temperature structure is not always clear. In many non metallic systems exhibiting first order transitions the volume change is so large that the crystal shatters as it undergoes the phase transformation. Many metallic systems exhibit first order transformations where there is nearly no volume change since the transformation is dominated by shear displacements. Martensitic transformations (MT) fall into this category.

Martensitic transformations have been studied extensively by metallurgists for nearly 100 years[3]. Electron diffraction and imaging has been widely used to probe the structure and microstructure of martensitic materials. The dynamical aspects of the transition have been probed primarily by use of ultrasonic techniques where the elastic constants can be measured. This is particularly valuable since martensitic transformations are frequently dominated by homogeneous lattice deformations which lead to anomalies in the measured elastic constants. Much effort has been extended to the study of pretransitional phenomena in martensitic alloys. One reason is that knowledge of the interactions that induce the pretransitional phenomena are vital to the understanding of the mechanism of the martensitic transformation and aid in predicting the low temperature structure of martensite. This is important in achieving the goal of enhancing and optimizing the technologically useful properties of these metallic solids.

In this review I shall discuss neutron scattering studies of pretransitional phenomena in beta-phase metals exhibiting martensitic transformations. Thermal neutron scattering is a unique probe of solids and the uniqueness is derived from its energy-momentum relation. The typical thermal neutron wavelength ($\lambda=1.5\text{\AA}$) is comparable to the size of the interatomic spacings in a solid and the thermal neutron velocity ($\sim 3\text{km/sec}$) is comparable to sound velocities in a solid. This makes the neutron the probe of choice in studying both the structure and dynamics of a solid. Because the energy of the neutron is quite low, typically 40 meV ($1\text{meV} = 8\text{cm}^{-1} = 11.6\text{K} = 0.24 \times 10^{12}\text{ Hz}$), inelastic measurements are straightforward to perform and only moderate resolution ($\Delta E/E \sim 10\%$) is needed. This is easily achieved with existing instrumentation. Neutron scattering is the only technique that allows one to measure the lattice vibrational modes over the entire Brillouin zone (BZ) of the solid. Below, the emphasis will be on the role of phonon softening in MT and a few examples of beta phase metals exhibiting typical premartensitic (PM) behavior will be discussed.

2. ALKALI METALS

The simplest materials exhibiting MT's are the Alkali metals: Li, Na, K, Rb and Cs. These exhibit nearly free electron behavior and the high temperature stable phase

has a simple BCC structure. Li and Na undergo a MT at a transformation temperature, T_M , near 70K and 35K, respectively. These transitions are highly hysteretic and the form of the Martensite microstructures are very dependent upon the sample, its purity and the manner and level of its thermal treatment. In K, Rb and Cs a temperature induced MT has not been observed. The low temperature structure of Li is now established to be a 9R structure although the transformation may not be complete [4]. In addition the low temperature phase exhibits extensive stacking faults which complicate the observed diffraction pattern and can lead to ambiguities in determining the structure [5]. The low temperature structure of Na is even less clear. It was thought to have an HCP structure, but a recent diffraction study observed a mixture of HCP, 9R and BCC [6].

The lattice dynamics of Li and Na has been studied several years ago [7,8]. It was noted that there was a large anisotropy in the two transverse modes propagating along the $[110]$ direction because of the anomalously low value of the elastic constant $c' = 1/2(c_{11} - c_{12})$. The ratio $c_{44}/c' = 5$ for Li and Na as determined from neutron scattering measurements. Zener noted this over 40 years ago [9] and suggested that this low value of c' is a result of the ion exchange interaction which renders the BCC lattice mechanically unstable with respect to a shear propagating along a $[110]$ direction and displacements along a perpendicular $\langle 1\bar{1}0 \rangle$ direction, referred to as $[110] - \langle 1\bar{1}0 \rangle$ mode. This phonon branch is called the Σ_4 branch for a BCC system. This is illustrated in Figure 1 and corresponds to sliding of $\{110\}$ planes along $\langle 1\bar{1}0 \rangle$ directions. The stacking sequence of $\{110\}$ planes in a BCC structure is ABABAB... (Fig 1a). Depending upon the wavelength of the mode propagating along $[110]$ a

Figure 1: Stacking sequences of $\{110\}$ planes of atoms in: (a) cubic BCC; (b) 9R structure composed of a shear plus $q=1/3[110]$ shuffle; (c) 3R structure; and (d) 7M structure with a $(5, -2)$ stacking.

variety of different structures can be generated as illustrated in Fig. 1. For the 9R structure of Li the wavevector is $1/3[110]$. As illustrated in Fig. 1b, however, this

alone will not give the 9R structure. A shear and an elongation along orthogonal directions is needed. This typifies many MT in that a modulation (frequently called a "shuffle") and a homogeneous deformation is needed to generate the Martensite structure.

Figure 2: Temperature dependence of [110] - $\langle 1\bar{1}0 \rangle$ transverse mode measured in Li by (a) Ernst et. al. (ref. 10) and (b) Smith (ref. 4).

There have been recent measurements of the temperature dependence of the $[\zeta\zeta 0]$ branch in Li by two different groups as shown in Fig. 2 [4,10]. These measurements were performed near room temperature and just above T_M . Both experiments exhibit a slight (10%) softening at the zone boundary and the amount of softening decreases as ζ decreases. (The curve in Fig. 2a shows a dip at $\zeta \approx 0.4$ but allowing for error bars this should not be considered significant). It should be noted that normal behavior in a weakly anharmonic solid shows an increase in phonon energy as the temperature decreases. The surprising feature is that no anomaly appears at $\zeta = 1/3$, which is expected because Li is known to transform into a 9R structure as discussed above. Also, a recent theory which describes the transformation and stacking faults selects $\zeta = 1/3$ as a special wavevector[11].

Neutron studies of the temperature dependence of this branch in Na produced conflicting results. One group reported a 5% decrease in the zone boundary phonon energy as T approached T_M [12], whereas another group recently reported no temperature dependence [5]. Could this be due to sample dependence? Clearly more work has to be done on the alkali metals before an understanding of the MT emerges.

3. TRANSITION METALS

Of the transition metals, Zr, Ti and Hf have stable BCC phases at elevated temperatures and undergo MT to an HCP phase at $T_M = 1155\text{K}$, 1133K , and 2023K ,

respectively. Under pressure Zr and Ti transform to the ω -phase with typical pressures of $p \sim 4$ GPa. The metallic alloys, Zr_xNb_{1-x} and Ti_xMo_{1-x} possess ω -like features if the alloys are quenched from high temperatures to room temperature. The state is usually a mixed phase and the BCC to ω -phase is not reversible with temperature.

Figure 3: Phonon dispersion curves of (a) BCC Zr from ref. 14 and (b) BCC $Zr_{0.8}Nb_{0.2}$ from ref. 16.

There have been several lattice dynamical studies of BCC Zr [13,14], Ti [14] and Zr_xNb_{1-x} alloy [15,16]]. Figure 3a shows the recent measurements by Petry et al. [14] of Zr measured in the high temperature beta-phase. The results are in substantial agreement with the earlier measurements by Stassis et al. [13]. There are two features of special interest: 1) It is seen that the $[\zeta\zeta0] - \langle 110 \rangle$ mode is particularly low in energy compared to the other branches. This mode corresponds to the shearing of the (110) planes discussed in Fig. 1. The temperature dependence of this branch shows that the phonon energy at the zone boundary decreases by $\sim 40\%$ between $T=1800K$ and $1168K$, just above T_M . Similar features were observed for Ti [14]. The displacements of the mode for the zone boundary phonon correspond to successive AB plane (see Fig. 1a) moving in opposite directions along $[110]$. As in the 9R structure, this alone is not sufficient for the HCP phase. There also has to be an adjustment of the atomic positions within the (1,1,0) plane to give the hexagonal symmetry. 2) The other interesting feature in the lattice dynamics is the dip along the $[\zeta\zeta\zeta]$ branch at $\xi=2/3$. The atomic displacements associated with this mode forces the collapse of two adjacent (111) planes while every third plane remains fixed. These are the precise displacements for the formation of ω -phase structures. The phonon energies are temperature independent and their dependence on pressure has not been measured. The complete phonon dispersion curve was also measured for the alloy $Zr_{0.8}Nb_{0.2}$ at room temperature and is shown in Fig. 3b [16]. An additional

symmetry direction is probed, the $[\bar{2}11]$ direction which is perpendicular to $[111]$. Note that the transverse $2/3[\bar{2}11]$ mode with polarization along $[111]$ is the identical mode as the longitudinal $2/3[111]$. The phonon dispersion curves along most symmetry directions are nearly identical for both pure Zr and $Zr_{0.8}Nb_{0.2}$. Both show that at $2/3[111]$ the phonon dispersion curve extends down to very low energies. The differences between the two systems is that in the pure system there is no elastic scattering at the $2/3[111]$, the F point [14]. At this point, energy scans reveal a very broad width so the mode can be viewed as overdamped. In the alloy there is intense elastic scattering arising from the ω -like embryos that are present in the alloy [15,16]. In the pure system embryos of the ω -phase are not formed. The alloy shows anomalous features along the $[\bar{2}11]$ symmetry direction. The transverse branch splits into two modes so there seems to be an excess of modes [16,17]. This has been interpreted as due to a local breaking of symmetry by the ω -like embryos and the extra mode arises from the long wavelength fluctuations associated with the short range ordered ω -embryos.

Recently there have been some first principles calculations of the phonon energies using the frozen phonon approach [18]. The total forces acting on individual atoms in a distorted crystal is separated into the ionic part and the electronic part by taking into account the electronic band structure. It was shown that the restoring forces for the phonon distortion in Zr is very weak and highly anharmonic. If the distortion is large enough the crystal will transform in the ω -phase. The calculations also showed that the BCC phase is unstable to a condensation of the transverse $[110] - \langle 1\bar{1}0 \rangle$ zone boundary acoustic phonon. The BCC phase is stabilized by the anharmonic interactions and as the temperature decreases the anharmonic effects decrease and the transformation to the HCP phase occurs. It is evident now that first principles calculations can be performed and explain the structural instabilities in simple solids.

4. NI BASED ALLOYS

4.1 $Ni_xTi_{1-x}(Fe)$

Ni_xTi_{1-x} is one of the most widely studied Ni based alloys mainly because it exhibits remarkable shape memory effects associated with its MT. The determination of the low temperature structure and the understanding of the phase transformation remained elusive for many years because there is an intervening premartensitic (PM) phase which greatly complicates the diffraction patterns used to determine the structure. A breakthrough occurred when it was shown that by adding a few percent of Fe to Ni_xTi_{1-x} , T_M is suppressed and the PM phase is readily observable over a relatively wide temperature range [19].

The lattice dynamics of Ni_xTi_{1-x} has been studied [20,21] and an anomalously low $[\zeta\bar{\zeta}0] - \langle 110 \rangle$ transverse branch observed with a dip at $\zeta=1/3$. Figure 4 shows equi-intensity contours of the scattered neutrons for the $Ni_{46.8}Ti_{50}Fe_{3.2}$ alloy measured in the cubic phase at $T=350K$ and $T=240K$ which is just above the premartensitic transformation temperature, $T_{PM}=232K$. It is readily seen that the mode decreases in energy around $\zeta=1/3$ as T_{PM} is approached. As T decreases below T_{PM} elastic

satellite peaks appear which have a strange property. They are slightly incommensurate and their deviation from commensurability changes from one BZ to another [22]. As the momentum transfer increases, the degree of incommensurability increases. Also the direction of the incommensurate wavevector has a "swirl" pattern as shown in Fig. 5 where the arrow indicates the

Figure 4: Equi-intensity contours measured in $\text{Ni}_{46.8}\text{Ti}_{50}\text{Fe}_{3.2}$ for the $[\xi\xi0] - \langle 110 \rangle$ phonon branch at (a) $T_{\text{PM}} < T = 350\text{K}$ and (b) $T_{\text{PM}} < T = 240\text{K}$. $T_{\text{PM}} = 232\text{K}$.

Figure 5: the $[001]$ - zone satellites observed in $\text{Ni}_{46.8}\text{Ti}_{50}\text{Fe}_{3.2}$ at $T_{\text{PM}} > T = 226\text{K}$. Only the directions are given. The length of the arrows are not significant. (from ref. 22).

direction of the satellite peak from the commensurate $1/3$ position. This peculiar variation precludes any description as a simple charge or mass density wave. One idea for interpreting this behavior used a "ghost lattice" where the satellite peaks have shifted to their positions determined by the low temperature structure but the fundamental Bragg peaks remained at their cubic position [23]. Fuchizaki and

Yamada [24] developed a model where fluctuations of the embryos of the low temperature phase couple strongly to the strain and qualitatively reproduced the observed results. Folkins and Walker [25] used a more complete Landau expansion of the free energy, but also included a first order coupling of the strain to the order parameter. They could quantitatively reproduce Fig. 5. These peculiar features of satellites of the modulated phase should be present in any system where there is a strong coupling between the order parameter and the strain field. Most materials exhibiting MT possess this property. Similar features have already been observed in AuCd [26] alloys by x-ray diffraction and in recent measurements on $\text{Ni}_x\text{Al}_{1-x}$ alloys [27].

4.2 $\text{Ni}_x\text{Al}_{1-x}$

$\text{Ni}_x\text{Al}_{1-x}$ is another extensively studied alloy which undergoes a MT whose temperature depends critically on composition. In fact, it is still not understood what microscopic quantity is varying so rapidly that a 1% change in composition can change T_M by nearly 200K [28]. Practically, this demonstrates the need to have extremely well prepared samples with no concentration gradients. It also makes it difficult to compare measurements made with different samples because the composition has to be known very precisely. Small differences in x can lead to drastically different behavior.

Recently there has been an extensive study of the cubic phase of $\text{Ni}_x\text{Al}_{1-x}$ combining electron diffraction and elastic and inelastic neutron scattering measurements performed on samples cut from the same large single crystal [29]. The electron diffraction will be discussed by Tanner et al at this conference [30]. $\text{Ni}_x\text{Al}_{1-x}$ transforms martensitically from a cubic CsCl type structure to a lower symmetry for $60 < x < 68$ with a transition temperature $10\text{K} < T_M < 400\text{K}$ [28]. The phonon dispersion curve of $\text{Ni}_{62.5}\text{Al}_{37.5}$ have been measured along several symmetry directions as shown in Fig. 6. Most branches behave normally except for the lowest lying

Figure 6: The phonon dispersion curve measured along several symmetry directions for $\text{Ni}_{62.5}\text{Al}_{37.5}$. The hatched regions along the abscissa indicate elastic diffuse scattering:

transverse $[\zeta\zeta 0] - \langle 1\bar{1}0 \rangle$ branch. This branch has been studied in detail as a function of composition and temperature [31,32]. Figure 7 shows the dispersion curve for this branch for 4 different compositions. It is seen that as the Ni composition increases, the mode gets softer. This is in accord with the elastic constant data which showed

Figure 7: The $[110] - \langle 1\bar{1}0 \rangle$ phonon branch measured in $\text{Ni}_x\text{Al}_{1-x}$ for $x=50, 58, 62.5$, and 63.9 at. %. The $[110] - \langle 001 \rangle$ transverse branch for $x=58$ is also shown.

that c' decrease with increasing x . Another feature is that there are kinks in the dispersion curve whose position in ζ varies with composition. As x increases the anomaly moves to small ζ -values. Of the 4 compositions shown in Fig. 7 only $x=62.5$ and 63.9 exhibit a MT near 80K and 320K , respectively. The temperature dependence of this branch is shown for $x=62.5$ in Fig 8a. It is seen that the anomaly deepens near $\zeta=1/6$ on cooling. In the lower portion we show the elastic scattering measured along the same ζ -direction as the phonon branch. A well defined elastic peak develops at $\zeta=1/6$, which is the same wavevector as the phonon anomaly, and its intensity increases as T decreases. The reciprocal of the elastic diffuse scattering varies as the $\omega^4(\zeta=1/6)$. This behavior is what is expected from defect induced diffuse scattering which, near the zone center, is the well known Huang scattering [33]. The elastic diffuse scattering observed in Fig 8b has also been observed in the electron diffraction study of the same alloy [29]. In the high resolution image of the diffraction pattern there exists regions where the CsCl structure is perturbed. The atomic displacements associated with this distortion give rise to the peak in the diffuse scattering.

At low temperatures $\text{Ni}_{62.5}\text{Al}_{37.5}$ transforms into a 7M structure [34]. This structure is derived from a $(5,-2)$ stacking of $(1,1,0)$ planes since it corresponds to a shear of 5 planes along the $[110]$ direction and then 2 planes in the opposite $[\bar{1}\bar{1}0]$ direction as shown in Fig. 1d. The diffuse scattering changes abruptly from a peak

near $\zeta=1/6$ to a series of Bragg peaks near $\zeta=1/7$. However the peaks are not equally spaced nor are they exactly at $\zeta=1/7$. This interesting feature is similar to the observations in NiTi and is discussed in more detail by Noda et. al. at this conference [27].

Figure 8: (a) the temperature dependence of the $[110] - \langle 1\bar{1}0 \rangle$ phonon branch for $\text{Ni}_{62.5}\text{Al}_{37.5}$. (b) The temperature and q dependence of the elastic scattering associated with the phonon branch (from ref. 32).

Stress is known to play an important role in MT. It was shown that uniaxial tension can induce the 7M structure in $\text{Ni}_{62.5}\text{Al}_{37.5}$ at room temperature [34]. Tanner et al used electron diffraction to study the stress induced 7M phase at room temperature which is formed at tips of cracks spontaneously generated at the thin edges of foil specimens [27]. He clearly sees the (5,-2) stacking. Recently we have applied a uniaxial compressional stress to the single crystal of $\text{Ni}_{62.5}\text{Al}_{37.5}$ at room

Figure 9: The low energy portion of the $[110] - \langle 1\bar{1}0 \rangle$ phonon branch of $\text{Ni}_{62.5}\text{Al}_{37.5}$ measured at room temperature for zero stress (o) and a compressive stress applied along the $[001]$ direction (•).

temperature. The stress was applied along the [001] direction and the [110] - $\langle 110 \rangle$ branch was measured. Figure 9 shows the behavior of this branch at zero stress and applied stress. It is seen that the stress reduces the phonon energy of this branch, mostly in the region of the anomaly. Experiments are now planned to probe the temperature dependence as well as varying stress to map out the phase diagram in more detail.

5. CONCLUSIONS

In this review I have shown how measurements of phonon dispersion curves by inelastic neutron scattering exhibit premartensitic effects well above T_M . In many materials exhibiting MT a modulation of the lattice and a homogeneous distortion is necessary to transform the crystal from the high temperature phase to the low temperature structure. These modulations frequently occur with wavevectors different from zero and the lattice dynamics can only be probed by neutron scattering techniques.

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