ANALYTICAL ULTRASONICS FOR CHARACTERIZATION OF METALLURGICAL MICROSTRUCTURES AND TRANSFORMATIONS

Moshe Rosen
The Johns Hopkins University
Baltimore, Maryland 21218

Application of contact (piezoelectric) and noncontact (laser generation and detection) ultrasonic techniques for dynamic investigation of precipitation hardening processes in aluminum alloys, as well as crystallization and phase transformation in rapidly solidified amorphous and microcrystalline alloys will be discussed. From the variations of the sound velocity and attenuation the precipitation mechanism and kinetics were determined. In addition, a correlation was established between the observed changes in the velocity and attenuation and the mechanical properties of age-hardenable aluminum alloys. The behavior of the elastic moduli, determined ultrasonically, were found to be sensitive to relaxation, crystallization and phase decomposition phenomena in rapidly solidified metallic glasses. Analytical ultrasonics enables determination of the activation energies and growth parameters of the reactions. Therefrom theoretical models can be constructed to explain the changes in mechanical and physical properties upon heat treatment of glassy alloys. The composition dependence of the elastic moduli in amorphous Cu-Zr alloys was found to be related to the glass transition temperature, and consequently to the glass forming ability of these alloys. Dynamic ultrasonic analysis was found to be feasible for on-line, real-time, monitoring of metallurgical processes.

INTRODUCTION

Ultrasonic nondestructive evaluation has traditionally been concerned with the search and location of flaws in materials structures and the determination of their distribution and orientation. A considerable body of knowledge has also been accumulated from ultrasonic scattering studies in assessing grain size and orientation effects in materials. Recently, it has become widely recognized that ultrasonic measurements can be used to characterize materials structures and hence properties such as strength, toughness, effect of residual stresses so as to supplement, or even replace, the conventional destructive techniques employed in metallurgy. Ultrasonic nondestructive characterization offers distinct advantages in that materials properties can be verified on actual components of engineering structures. The scientific literature is extremely scarce in dynamic nondestructive characterization (NDC) whereby ultrasonic techniques are applied to on-line, real-time, monitoring of microstructures for the control of metallurgical processes. Inherent difficulties related to these types of measurement methodologies include operation at elevated temperatures and in hostile environments. Furthermore, there are little basic data available on the relationship between the metallurgical microstructures and measured ultrasonic responses. Therefore, the full potential of ultrasonic techniques in this field is yet to be realized.

Recent studies have addressed the specific area of dynamic, real-time nondestructive characterization of metallurgical microstructures, e.g., precipitation hardening of aluminum alloys and crystallization of amorphous alloys (ref. 1-5). Conventional bulk ultrasonic techniques were adapted for application to continuously monitor changes of the microstructure and the effect on physical properties. However, in attempting to dynamically characterize metallurgical processes in amorphous ribbons or structurally modified thin surface layers, the conventional ultrasonic techniques were found to be inadequate. Thus, a new approach for contactless generation and detection of acoustic waves, using a laser generation and laser interferometric detection system, was developed (ref. 6).

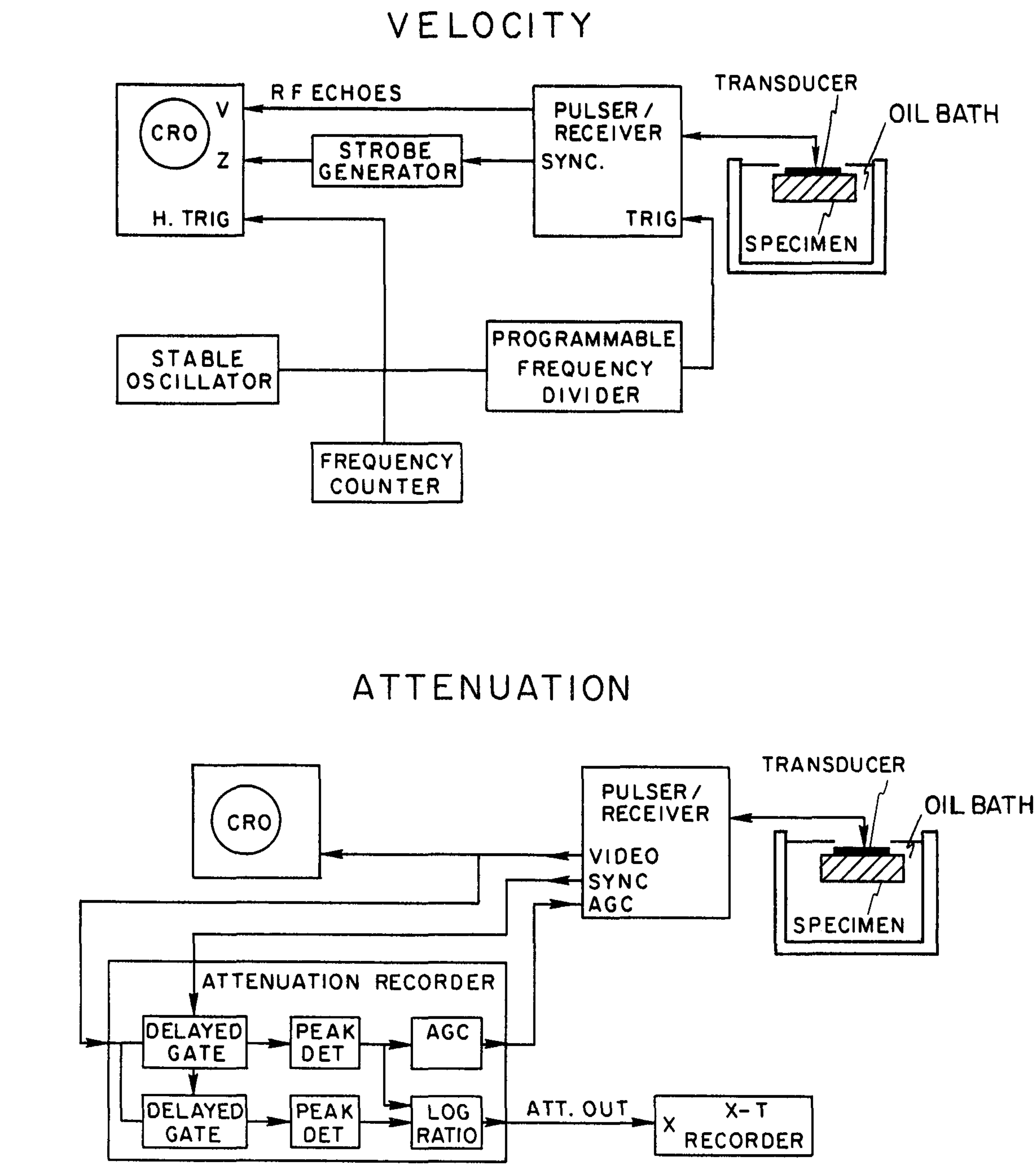
ULTRASONIC WAVES: GENERATION AND DETECTION TECHNIQUES

The relationship between experimentally determined sound wave velocities (longitudinal and transverse) and the elastic moduli, is expressed in terms of the equations of motion of elastic waves in a solid, and Hooke's law for an isotropic medium.

Thermodynamic formalism shows that a solid should exhibit an anomalous elastic behavior ("softening effect") in the vicinity of a phase transformation point. Anomalous changes in the elastic moduli are expected to occur, to a different degree and magnitude, in the proximity of metallurgical reactions involving precipitation processes, segregation of solute, magnetoelastic effects accompanying magnetic ordering and other phenomena.

The propagating sound waves in the solid undergo a series of loss processes due to scattering by reflection and refraction from grain and phase boundaries, thermoelastic losses including elastic anisotropy effects in polycrystals (Zener effect), as well as attenuation of the sound wave energy due to interaction of the proagating wave with electrons and phonons. Interaction with dislocations in the crystal lattice is also responsible for the observed sound-wave damping effects. In instances where dislocation movement plays a prominent role, e.g., in martensitic phase transformations, the behavior of the ultrasonic attenuation may contain important information with respect to the transformation mechanism. During precipitation hardening of 2024 aluminum alloy the main contributions to observed changes in attenuation (ref. 1) were found to be due to geometric scattering effects by newly formed precipitates of a certain critical size distribution, as well as to resonant interaction between the propagating sound waves and the dislocation loops generated around precipitates.

For bulk ultrasonics, where the wavelength of the propagating waves is much smaller than the specimen dimensions, the velocity and attenuation of ultrasound in specimens can be determined through the use of a pulse-echo overlap technique (ref. 7). The ultrasonic transit time, between the opposite faces of a flat and parallel sample, is determined by overlapping two successive members of an echo train on an oscilloscope display. This method, by stroboscopic identification, allows the time interval between pulses to be determined to within 1 part in 10^4. The logarithmic decay of the amplitude of successive pulses determines the ultrasonic attenuation. The apparatus for measuring both velocity and attenuation is shown in figure 1.



ATTENUATION AND VELOCITY MEASURING SYSTEMS

Figure 1.

Dynamic measurements of sound velocity and ultrasonic attenuation, during metallurgical changes in the temperature interval between the ambient and 250^{o} C, were performed while the specimen and the ultrasonic sensors were in direct contact in an oil bath. For static ultrasonic measurements, carried out at room temperature, with the purpose of correlating ultrasonic data with mechanical properties, both the samples and the sensors were immersed in a distilled-water tank at a constant separation. Careful adjustments were made to ascertain an exponential decay of the amplitude of successive echoes, combined with a maximal number of echoes in the pulse train.

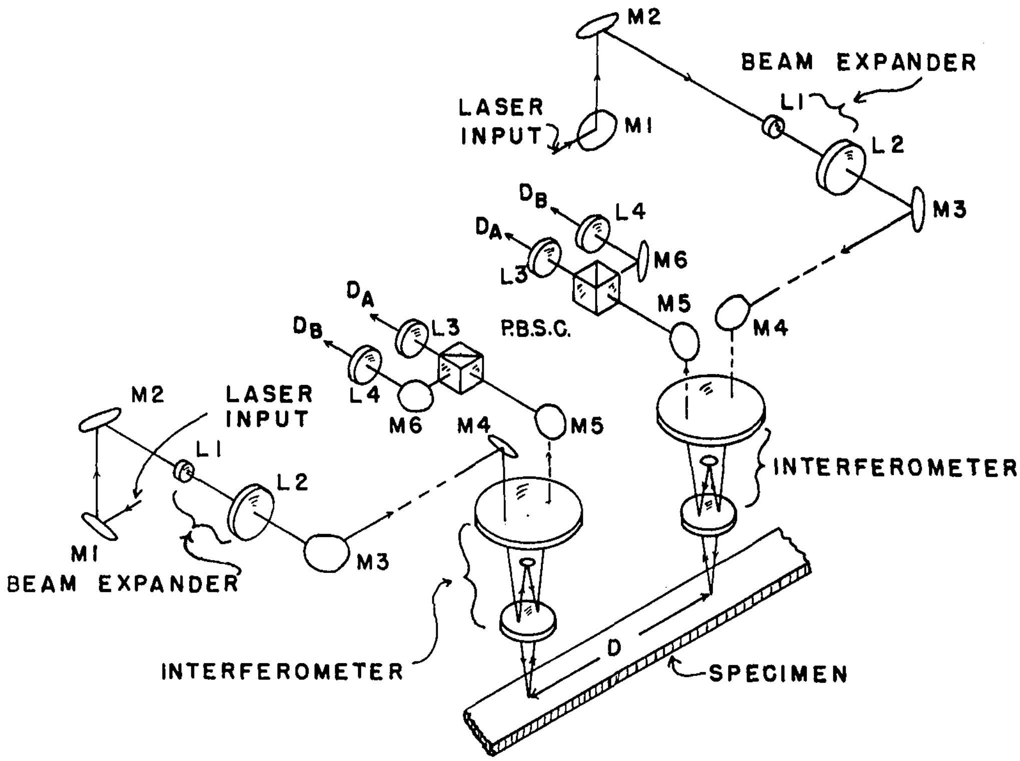
The growing interest in metallic glasses has created the need for the determination of their physical and mechanical properties. Some of these properties, especially the mechanical ones, cannot be measured conveniently by traditional ultrasonic methods as the high cooling rates required to form metallic glasses restrict their physical shapes to shallow layers or thin ribbons. Alternate measurement methodology needed to be developed in order to accurately assess these properties. One of the techniques employs a coil to magnetostrictively launch and detect extensional waves in a ribbon or magnetic material, called the "driver." The specimen can be coupled to the end of the driver, producing an echo pattern from which the extensional velocity in the material can be derived by a pulse-superposition technique (ref. 7). A pulse-receiver was constructed to feed high current pulses to the coil and to amplify the electrical signals produced in the coil by the acoustic waves.

The noncontact feature of both generation and detection of ultrasonic waves can be very advantageous in situations requiring physical separation between the measuring system and the material under investigation, e.g., when high temperatures or hostile atmospheres are involved. Furthermore, the contactless generation and detection precludes interaction with, and modification of, the wave propagation pattern under study. In addition, laser generation of acoustic waves yields a wide variety of propagation modes (longitudinal, transverse and plate modes, Rayleigh waves) over a wide frequency range, thus enhancing the amount of information obtained from a single measurement.

Compressive stress waves that propagate in a material can be generated by transient loads applied by rapid energy transfer from a single-pulse Q-switched high-energy Nd:Yag laser, (fig. 2). Propagation of ultrasonic waves in a medium causes surface displacements on the material that can be measured optically by exploiting the phase shift of an optical beam reflected from the surface of the material. When the reflected beam is combined with a reference optical beam, from a helium-neon laser, optical phase changes are converted into amplitude phase changes that are detectable by a sensitive photodiode. These variations in amplitude are proportional to the surface displacements on the specimen. Potential problems arising from the fact that phase changes also result from relative motion among optical components of the system and from temperature and pressure fluctuations of the ambient air, are prevented by appropriate design of the interferometer. An optical scheme due to Fizeau is particularly suitable for our specific purpose. Two optical probes are separated to allow accurate measurements of travel time of an ultrasonic wave in the material over a well-defined distance. Furthermore, the variation in magnitude of the surface displacements detected by the two interferometers determines the ultrasonic attenuation in the material. Thus, both velocity and attenuation can be measured simultaneously, fig. 3. The interferometer was built at Johns Hopkins University by Dr. Harvey Palmer.

Compared with other sensors, optical interferometers offer several advantages.

Overall Optical System



DUAL LASER INTERFEROMETER.

Figure 2.

SOUND VELOCITY MEASUREMENT SYSTEM

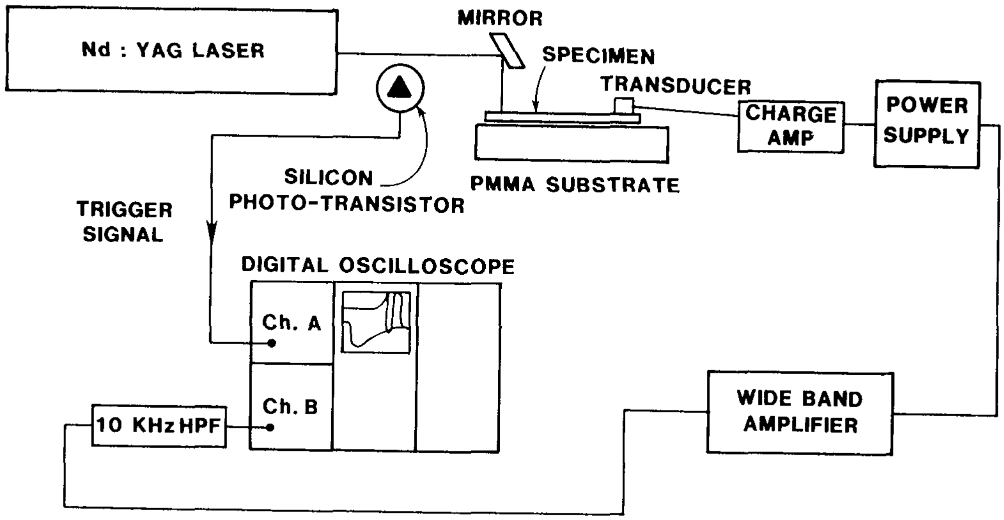


Figure 3.

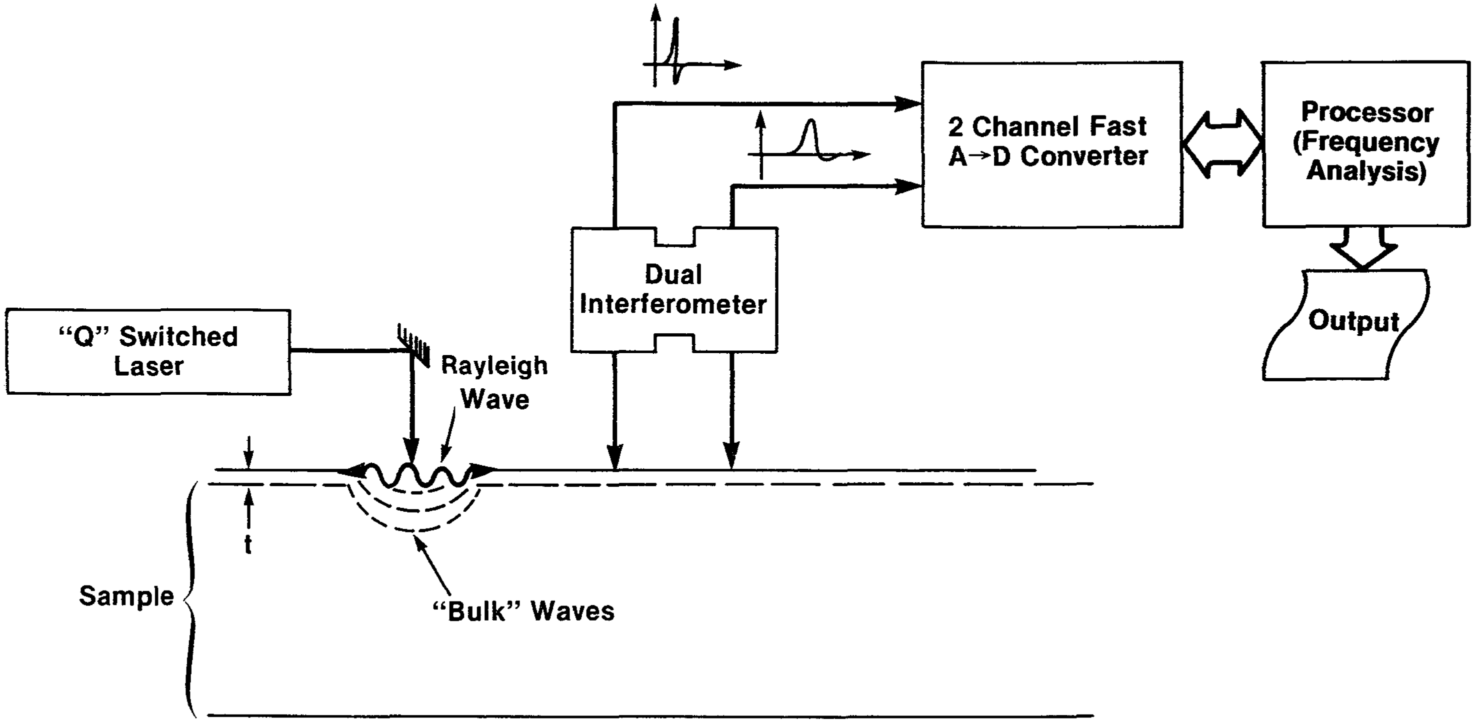
The sensitive area can be made very small, a few micrometers in diameter, for highly localized measurements. Consequently, the surface of the specimen does not necessarily have to be flat optically. The highly focused optical beams permit utilization of specimens with conventionally machined surfaces. The measured quantity is linear displacement. Independent methods of absolute calibration are applicable. Bandwidth, determining the fidelity of reproduction of signal waveforms, is not limited by the character of the transduction process but by the electronics of the interferometer detectors. Therefore, performance can largely exceed that of conventional piezoelectric transducers. Small signal resolution and bandwidth are related, thus linear displacements of a few angstroms are detectable at 7 MHz bandwidth.

Laser pulse irradiation produces a stress pulse of short duration (15 ns) and relatively high amplitude (up to 200 mJ power) making the investigation of very thin (about 20 µm) and highly attenuating specimens possible. Since a multitude of acoustic wave propagation modes is generated the dual interferometer provides important information concerning the elastic and inelastic properties of the material under static or dynamic conditions.

The contactless generation and detection of acoustic waves is extremely advantageous because specimens can be studied while they are subjected to thermomechanical processing under adverse environmental conditions. No transducer protection (e.g., from elevated temperatures) is necessary, and the detected ultrasonic waveform is unperturbed by extraneous effects due to physical contact between specimen and transducer. Data acquisition is straightforward and real-time or post-test analysis is feasible. The capability to obtain the frequency dependence of both sound velocity (in dispersive regimes) and ultrasonic attenuation offers new opportunities in solid state and physical metallurgy research where the elastic properties and acoustic energy absorption play a prominent role in the characterization of the processes.

Laser and electron beam irradiation techniques are being extensively applied for the modification of surface properties of metallic structures (ref. 9, 10). The nature of the modified surface zones is not amenable to conventional nondestructive characterization. However, the analysis of Rayleigh wave velocities, and the determination of the elastic moduli, may lead to a better understanding of the properties of the modified surface layers. Laser or piezoelectrically generated Rayleigh surface waves probe preferentially the near-surface region of the sample, and are extremely sensitive to variations in the elastic properties and ultrasonic attenuation of the material medium, figure 4. The extent of penetration of the Rayleigh surface wave normal to the surface of the sample depends on the frequency of the propagating Rayleigh waves and the exponential decay of their intensity. For a typical metallic phase, the Rayleigh wave velocity, V , is about 3000 ms^{-1}. For a frequency of 10 MHz, the Rayleigh wavelength is, therefore, 300 µm. Theory shows that 90 percent of the energy of the Rayleigh wave is contained within one wavelength from the surface. Thus, the sub-surface region can be monitored with a high degree of accuracy. Moreover, Fourier analysis of the frequency content of the Rayleigh waves, when the dual-laser interferometer is used to contactlessly detect the propagating surface waves, allows the precise determination of the thickness of the surface layer. This gauging procedure is possible because of the unique property of the Rayleigh wave velocity that is independent of frequency.

Two properties of the Rayleigh surface wave make its detection possible by optical means. One, is the surface microcorrugation (microdistortions) as the Rayleigh wave propagates through the material medium. Second, is the periodic



- **Wideband Ultrasonic Pulse Generated by Laser Pulse.**
- **Vertical Displacement v.s. Time is Measured at Two Different Points by Two Wide Band Optical Detectors (Interferometers).**
- **Frequency Analysis of the Two Pulses Gives Attenuation v.s. Frequency and Velocity v.s. Frequency Curves.**
- **Since the Penetration of Rayleigh Waves is Frequency Dependent, the Thickness t of the Amorphous Layer can be Evaluated From These Curves.**

RAYLEIGH WAVE VELOCITY MEASUREMENTS IN MICROSTRUCTURALLY MODIFIED LAYERS EMPLOYING LASER GENERATION AND LASER-INTERFEROMETRIC DETECTION OF ULTRASONIC WAVES

Figure 4.

variation of the index of refraction while the wave propagates on the surface. By means of optical interferometry, e.g., dual Fizeau interferometer, these waves can easily be detected in a contactless fashion. Thus, the contactless generation and detection of Rayleigh waves enables one to dynamically study the near-surface modifications of the material while the samples are contained in a hostile environment, and are subjected to programmed thermomechanical treatments subsequent to laser or electron beam alloying and microstructural modification.

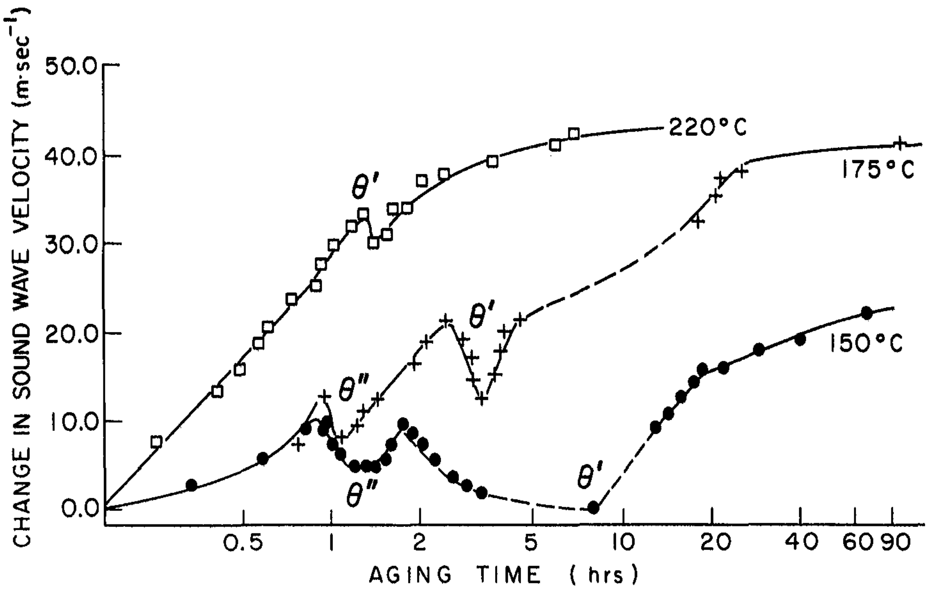
NONDESTRUCTIVE CHARACTERIZATION OF PRECIPITATION HARDENING PHENOMENA IN ALUMINUM ALLOYS

Precipitation hardening, or aging, is an important metallurgical process whereby the material's strength and hardness can be augmented, in several instances, by an order of magnitude. Precipitation hardening is a thermally-activated, diffusion-controlled, process of particular importance in copper, iron, and aluminum-base alloys. The improved mechanical and physical properties of these alloys depend, largely, on the microstructure, spacing, size, shape, and distribution of the precipitated particles, as well as on the degree of structural and crystallographic coherency of the particles with the matrix. For example, the precipitation hardening process in aluminum alloys involves formation of Guinier-Preston (G.P.) zones, θ'' and θ' particles that are generally coherent or semicoherent with the matrix and contribute to the strenghthening of the alloy. At higher temperatures incoherent particles form, that dramatically reduce the strength and hardening of the alloy. Of particular importance in aluminum-base alloys are the θ' particles, initially semicoherent with the matrix, that grow in size as the precipitation process progresses. At a certain critical size, when the stress fields surrounding these precipitates are maximal, dislocation rings (Orowan loops) form around them causing the coherency to be weakened. At this point overaging, or softening, begins.

Of great technological importance is the development of a nondestructive probe that will permit: (a) determination of the extent of matrix supersaturation in alloys prepared by new processes such as rapid solidification, and (b) real-time monitoring of the precipitation hardening process to determine particular events such as the instant when particles lose coherency with the matrix and overaging begins. Of course, solution of the latter problem implicitly addresses the former.

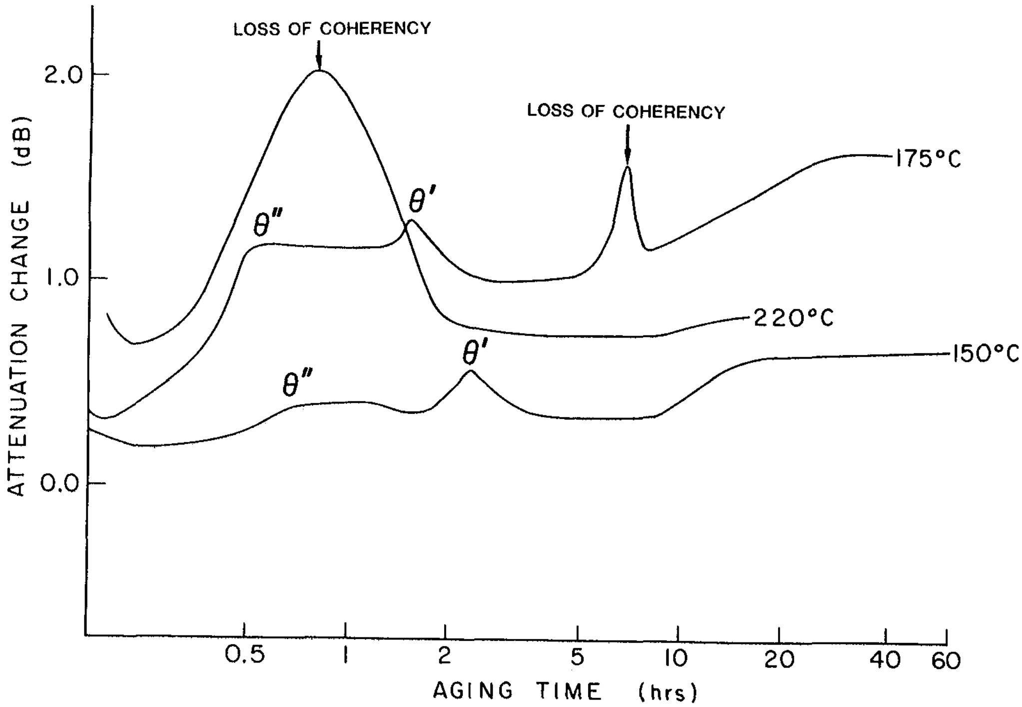
Figure 5 shows the time dependence of the changes in the sound velocity of 2219 aluminum alloy during isothermal aging at three temperatures. The nondestructive characterization of the aging process, as manifested in figure 5 and corroborated with optical and electron microscopy, hardness, strength, and eddy currents conductivity data, shows a series of dips that correspond to the maximal rate of formation of θ'' and θ' particles.

The prominent peaks in ultrasonic attenuation, figure 6, correspond to the peaks in hardness and represent the loss of coherency of θ' particles with the matrix and beginning of the softening process. The peaks in ultrasonic attenuation arise from an interaction between the acoustic vibrations of the ultrasonic waves propagating through the material and the interface dislocations surrounding the θ' particles. The strength of the interaction will depend on the total volume of the θ' particles, their size, shape, and distribution. The peak value of attenuation can be qualitatively related with the total volume of coherent precipitates that lost coherency with the matrix. The temperature dependence indicated by the shift in the



ULTRASONIC VELOCITY DURING THREE ISOTHERMAL HEAT TREATMENTS OF 2219 ALUMINUM ALLOY

Figure 5.



ULTRASONIC ATTENUATION DURING THREE ISOTHERMAL HEAT TREATMENTS OF 2219 ALUMINUM ALLOY

Figure 6.

attenuation peaks, figure 6, permits the determination of an activation energy that is found to be very close to that obtained from the shift of the hardness peaks (ref. 1). Corroborative evidence was obtained by means of electron microscopy and substantiates the proposed mechanism of coherency loss in this alloy.

This study demonstrates the feasibility of the ultrasonic NDC technique to determine the time of formation of the various precipitates, the kinetic parameters of the process, as well as to establish, by means of ultrasonic attenuation, the time when the alloy begins to suffer a loss of its mechanical properties due to overaging.

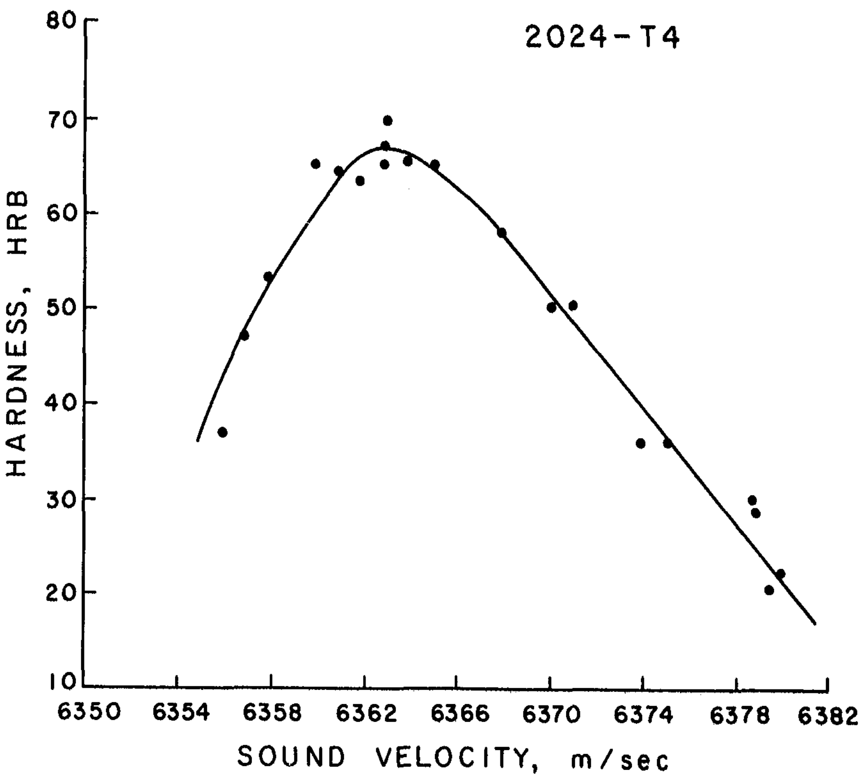
Nondestructive characterization measurements have been carried out on 160 Al-Cu alloy samples subjected to a series of different preaging heat treatments prior to thermal processing to different tempers. For each temper the maximum hardness of the alloy was found to correlate to a particular value of the sound wave velocity. In addition, ultrasonic attenuation was found to consistently decrease in a linear fashion as hardness is increased, figures 7 and 8. This investigation demonstrates the potency of ultrasonic NDC for in-process monitoring and control of the mechanical properties (hardness, strength) of age-hardenable aluminum alloys. The correlation suggests that within a range of thermomechanical treatments the hardness of the alloy can be uniquely determined by means of sound velocity and ultrasonic attenuation.

NDC OF CRYSTALLIZATION PROCESSES IN AMORPHOUS MATERIALS

Amorphous alloys, or glassy metals, are solids with frozen-in liquid structures. The absence of translational periodicity in the amorphous state along with the macroscopic compositional homogeneity are the main reasons for their improved proerties, e.g., high mechanical strength, good corrosion resistance, and excellent magnetic behavior. Their unusual mechanical, chemical, and physical properties have stimulated extensive scientific and technological interest. One serious problem in the processing and utilization of amorphous alloys that may limit their future technological applications is their low thermal stability. When thermomechanical conditions are appropriate, metallic glasses relax structurally and ultimately crystallize into more stable structures resulting in drastic variations in properties. The factors governing the thermal stability of these alloys and their effect on properties are not well understood. For example, upon crystallization, amorphous alloys undergo very large changes in the elastic (40 percent) and anelastic properties with accompanying reduction in plastic properties (embrittlement). For this reason, availability of a nondestructive ultrasonic characterization technique for both property determination and metallurgical process control can be extremely useful.

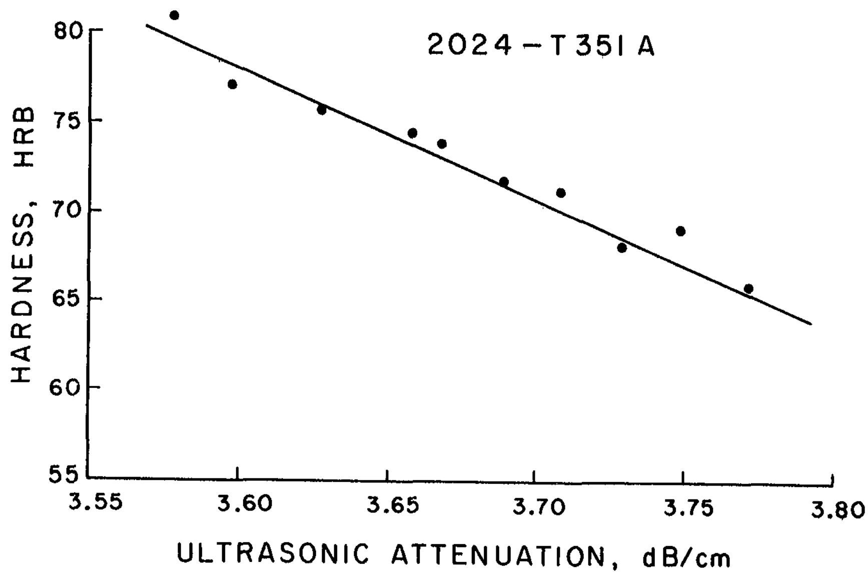
There is also ample evidence that microstructural modifications of near-surface layers, with concurrent improvements in properties, can be achieved by laser and electron beam irradiation of bulk metallic materials. Rapid melting and resolidification of a surface layer can produce refined structures and more homogeneous materials, or, in some instances, new microstructures and phases. Examples of the latter include extended solid solubility and formation of metallic glasses.

Ultrasonic NDC, in particular the contactless generation and detection of Rayleigh surface waves by laser interferometry, can be very useful for determination of the properties and geometrical dimensions of structurally modified layers while in the formative or post solidification stage.



VARIATION OF THE SOUND WAVE VELOCITY
WITH HARDENESS IN AGE HARDENED SAMPLES

Figure 7.



VARIATION OF THE ULTRASONIC ATTENTUATION
WITH HARDNESS IN AGE HARDENED SAMPLES

Figure 8.

Metallic glass ribbons of PdCuSi and CuZr alloys were prepared by the melt spinning technique. The samples were approximately 40μm thick and 1.25 mm wide. Ultrasonic, x-ray, and metallographic examination prior to the crystallization treatment revealed no crystallinity. Crystallization was performed in evacuated quartz capsules at predetermined isothermal holding temperatures. Optical metallography indicated that the crystallization process in PuCuSi ribbons is not homogeneous in the bulk. Two coexisting growth processes were observed. Frontal growth from the free surfaces of the ribbon towards the interior, and homogeneous nucleation and growth in the bulk.

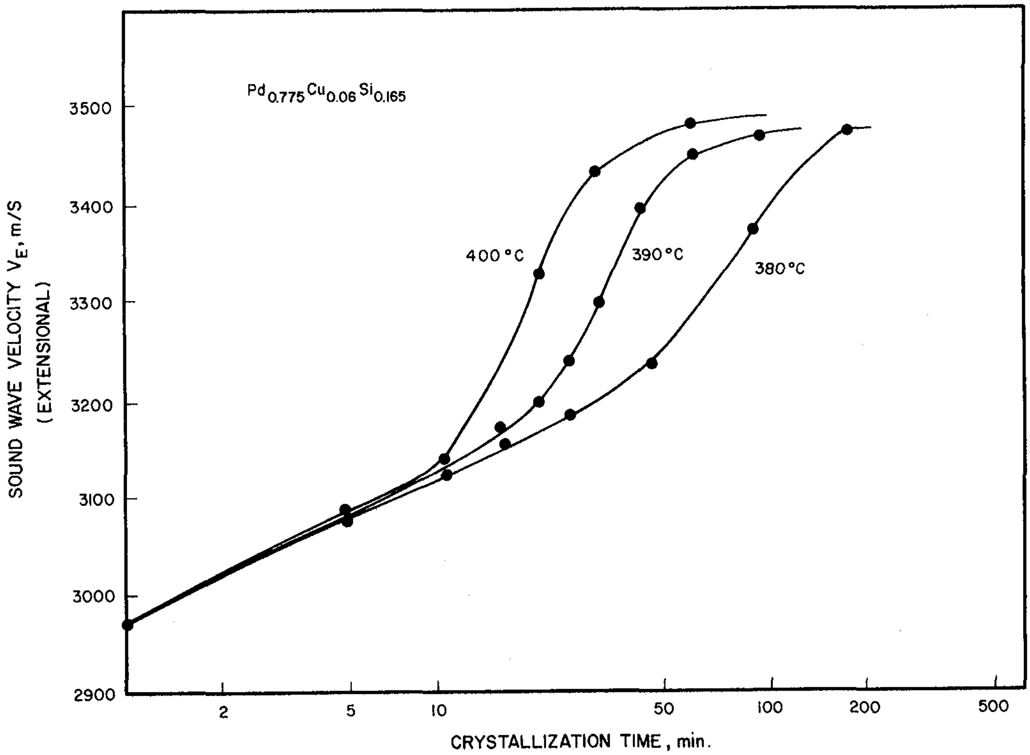
The velocity of the ultrasonic extentional waves was determined by measuring the transit time of a single pulse generated by a laser and detected by a piezoelectric quartz-crystal tranducer located at a distance of about 200 mm from the spot on the ribbon irradiated by the laser. The transient load was applied by rapid deposition of energy from a single-pulse of a Q-switched neodymium YAG laser wtih a wavelength of 1.06μm . The laser-pulse duration was 15 ns, and the pulse energy, for this specific series of measurements, was about 20 mJ. The elastic stress wave propagates along the specimen and may be detected by a piezoelectric transducer. The laser pulse was line-shaped to produce a nearly plane wave. A photodetector was used to trigger a transient pulse recorder as the laser pule was generated. Thus, the transit time of the extensional sound wave propagating along the ribbon could be determined to within better than 1 part in 10^4.

Figure 9 shows the variation of the extensional wave velocity, V as a function of crystallization time at three isothermal holding temperatures, 380, 390, and 400 ^{o}C. The initial sound-wave velocity, in the amorphous state, was found to be 2970 ms^{-1}, whereas in the fully crystallized state it reached an asymptotic value of 3490 ms^{-1}. Taking 10.52 and 10.69 g cm^{-3} as the density of the amorphous and crystalline $Pd_{77.5}\ Cu_6\ \ Si_{16.5}$ ', respectively, the Young moduli could be calculated. They were found to be 9.46 x 10^3 kg-mm^{-2} for the amorphous state and 13.27 x 10^3 kg-mm^{-2} for the crystalline state, i.e., E of about 40 percent. Figure 9 exhibits a relatively sharp increase in the sound velocity during the first five minutes of the isothermal holding. The variation of the extensional wave velocity with crytallization time manifests the dramatic changes in the elastic properties with an increase of about 40 percent in Young's modulus. The sigmoid curves represent the crystallization kinetics which are a thermally activated process. Thus, the kinetic parameters, i.e., activation energy and the growth regime parameters can be determined form the NDC data (ref. 3, 4). The obtained values were found to be in satisfactory agreement with calorimetric data (ref. 11) of activation energies for crystallization of amorphous alloys (ref. 12). Furthermore, the time dependence of the crystallized volume fraction was found to be compatible with a diffusion controlled growth mechanism (ref. 13, 14) for a plane boundary growth that is preceded by a rapid nucleation process (ref. 15).

The effect of copper content on the Young's modulus of ZrCu alloys in the crystalline and amorphous state indicates that the lowest value of the Young's modulus is for pure crystalline zirconium, whereas the highest value is for pure crystalline copper. A maximum and a minimum are found for alloys with compositions near the intermetallic compounds Zr_2Cu and ZrCu, respectively. The variation of the Young's modulus with composition of the amorphous alloys, between 30 and 70 percent copper, is a smooth S-curve with an inflection point at 50 a/o copper. It is conjectured that the amorphous state can be interpreted in terms of short range order structure where the mixing pairs Zr-Zr, Cu-Cu, and Cu-Zr are of equal probability at the composition of 50 a/o Cu. The inflection point on the Young's modulus versus composition curve will then represent the point for maximal configurational entropy of the amorphous alloy. Analysis of the temperature dependence of the sound wave velocity of Zr-Cu alloys in the composition range between 30 and 70 a/o Cu allowed determination of the activation energies of the diffusional processes governing the crytallization kinetics. The activation energy increases from 90 to 110 kcal/mole as copper content exceeeds 50 a/o. The change in crytallization mechanism as copper content exceeds 50 a/o may be related to the configurational entropy change as the population of Cu-Cu pairs increases. The relationship between the behavior of the Young's modulus as a function of composition, and the variation of the activation energy of the crystallization process with copper content is yet to be elucidated. It is believed that this relationship is of basic importance in the understanding of the transformation of the amorphous state into coexisting compounds upon isothermal crytallization (ref. 5).

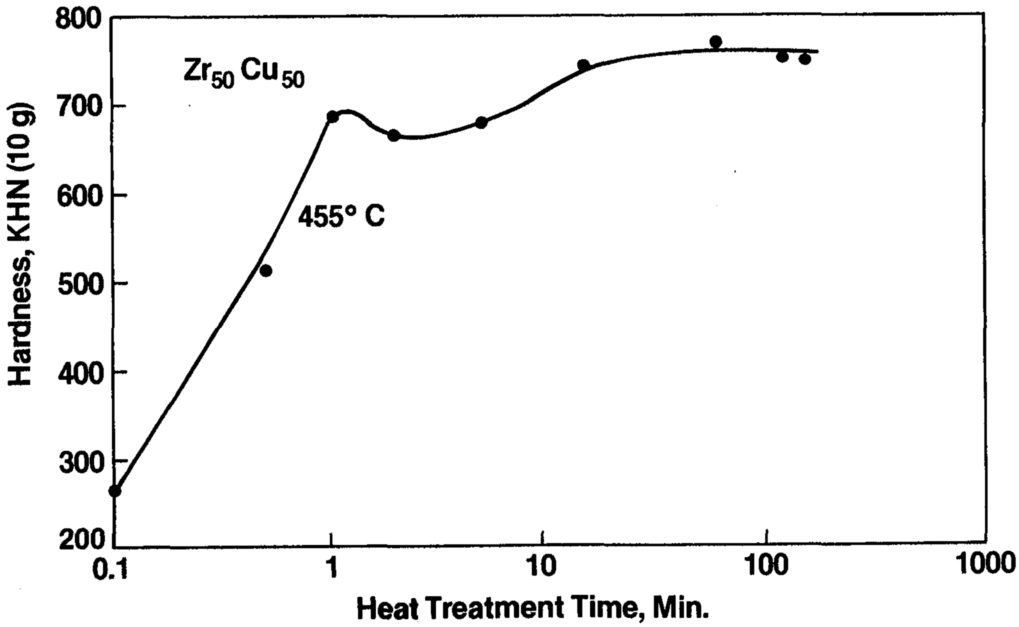
The initial increase in the extensional sound velocity, of about 3.7 percent, i.e., 7.4 percent in the Young's modulus of PdCuSi and a similar increase in the Zr-Cu amorphous alloys, appears to be related to a structural relaxation that precedes the crytallization process in the amorphous state (ref. 14, 16). Structural relaxation was found to be particularly enhanced in glasses obtained at high quenching rates, where greater structural disorder is to be expected (ref. 17). The lower elastic stiffness in the glassy state has been attributed to the interatomic displacements inherent in a disordered structure (ref. 18). The elastic stiffness should, therefore, increase with increased local ordering as a consequence of the structural relaxation. Microhardness measurements on the ribbon edge, perpendicular to the length vector of the ribbon, revealed a dramatic increase in hardness while the material is still in the amorphous state, before crystallization ensues, figure 10. The embrittlement mechanism in the amorphous alloy can be related to the removal of the quenched-in free volumes (vacancy clusters). Initially, the amorphous alloy has a disordered packing of the constituent atoms. The reduced coordination of the atom complexes, due to the presence of excess free volumes, allows shear transformations to be sustained. The free volumes are analogous to dislocation core segments. Consequently, the material is very ductile in the disordered amorphous state. Heat treatment in the amorphous region at temperatures below the glass transition temperature, T_g, or for relatively short periods of time at temperatures above T_g causes structural and compositional relaxation. The free volumes are redistributed by short range shuffling of the atoms, thus a more rigid close-packed atomic distribution of high coordination is achieved. The dense random packing of hard spheres (Bernal structure) leads to significant embrittlement of the amorphous phase.

The topological and compositional relaxation processes occurring in the amorphous state can be investigated by analyzing the behavior of the shear and compressional components of the ultrasonic attenuation, and their frequency dependence. From the frequency dependence of the ultrasonic attenuation, obtained by means of the dual-laser interferometer, the behavior of the bulk and shear viscosities can be characterized. Consequently, it is possible to establish the functional relationship between the appropriate relaxation times and the diffusional reactions leading to topological and compositional changes in the amorphous state. This specific phase of the investigation is currently in progress. It can be realized through the capability of the laser generation-laser interferometric detection of propagating sound waves, and the subsequent analysis of their frequency dependence within the framework of the viscoelastic theory.



VARIATION OF EXTENSIONAL WAVE VELOCITY IN PdCuSi DURING TRANSITION FROM AMORPHOUS TO CRYSTALLINE STATE

Figure 9.



VARIATION OF MICROHARDNESS DURING CRYSTALLIZATION OF AN AMORPHOUS ZrCu ALLOY

Figure 10.

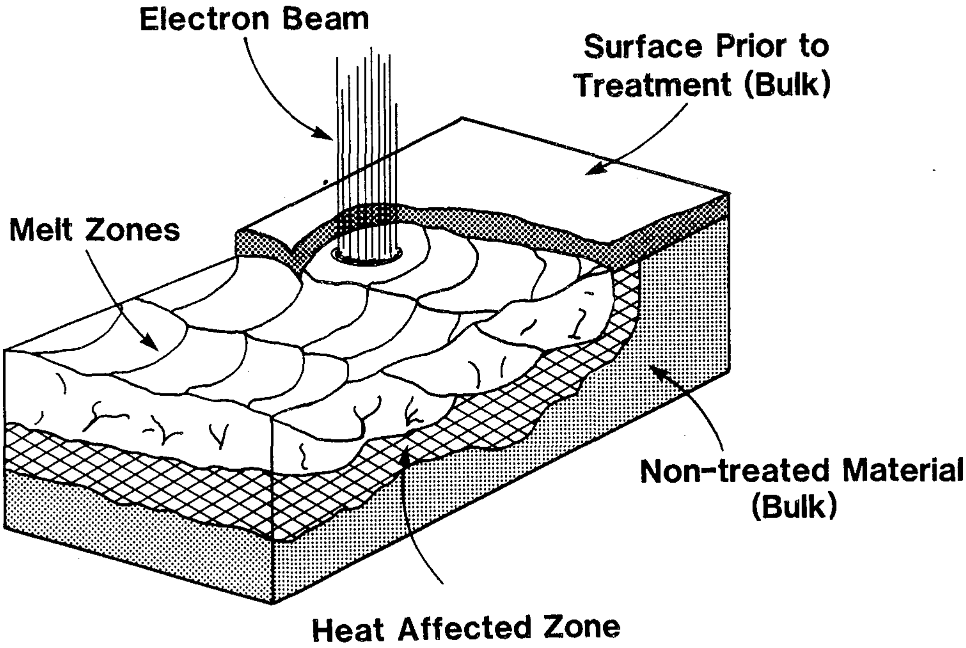
NDC OF MICROSTRUCTURALLY MODIFIED SURFACE LAYERS

High power lasers and electron beams have recently been applied for modifying metal surfaces and improving their physical, chemical, and mechanical properties (ref. 9, 10). Modification of surface properties may involve incorporation of alloying elements into the surface layer or localized thermomechanical treatment leading to formation of microstructures of desired characteristics. The focused laser or electron beam energy is deposited in such a fashion as to melt a thin layer while the bulk of the material provides the rapid quenching effect. Typical quenching rates may be of the order of $10^6 ks^{-1}$ or higher (fig. 11). Control of the developing microstructures and their characterization is crucial to the understanding of the operating mechanisms responsible for the near-surface reactions and for the exploitation of the great potential of this emerging technology for specific applications. Nondestructive characterization may be of prime importance for the on-line, real-time mechanical and physical evaluation of the surface properties, or as basic parameters for engineering design. Demands for safety and quality control have emphasized the lack of an acceptable NDE technique for quality assurance and process control. In a recent review (ref. 19) various NDE methods were discussed for application on surface coatings: electrical, magnetic, optical, and acoustic. Only those techniques based on acoustic properties showed sufficient promise for engineering applications.

Electron beam glazing has been applied in the course of the present research to develop modified metallurgical microstructures such as amorphous layers on crytalline substrates of PdCuSi (fig. 12), deposition of copper on 1100 aluminum samples followed by electron beam melting in an attempt to form an aluminum-copper surface layer on the aluminum bulk, and formation of metastable martensitic microstructures on a pearlitic bulk of AISI 1045 steel (fig. 13).

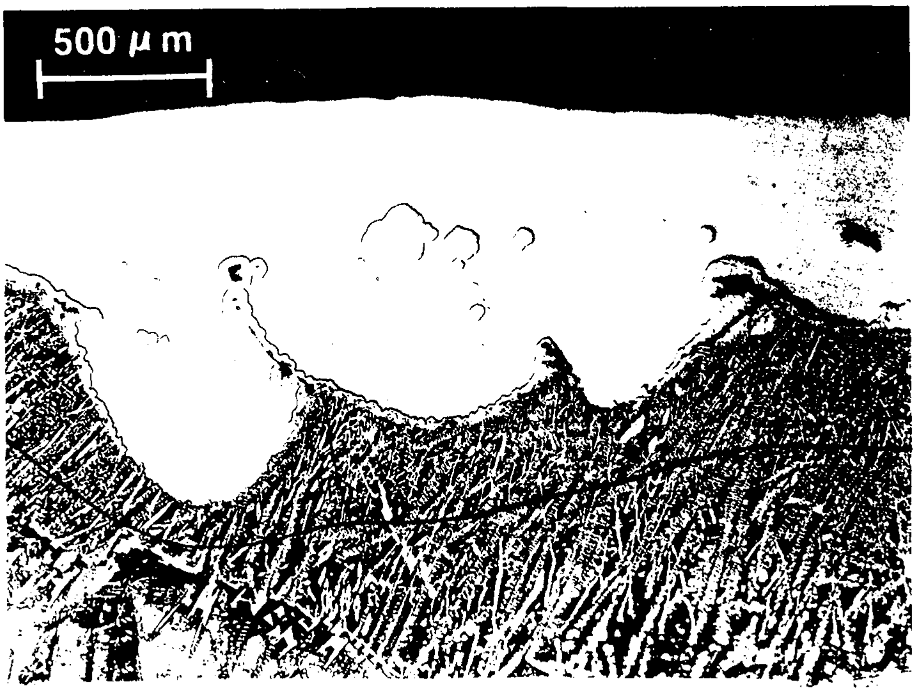
Figure 12 shows an amorphous layer of 1 mm average depth produced by electron beam melting and rapid solidification of a dendritic crystalline substrate. The heat affected zone is apparent as well as the grain structure of the original polycrystalline material.

Ultrasonic NDC of the thermally modified peripheral layer was determined by means of Rayleigh surface waves, using the device depicted in figure 14. As mentioned previously, the Rayleigh wave velocity is frequency independent. However, the extent of penetration of Rayleigh waves normal to the material surface is frequency dependent. Frequency analysis of the apparent Rayleigh velocities enables determination of the elastic properties of the layer, and the gauging of its average thickness. The Rayleigh surface wave velocity was measured by means of a wedge device (fig. 14) which converts longitudinal waves into Rayleigh waves, and vice versa. In the case of an amorphous PdCuSi layer, in which the Rayleigh surface wave velocity is about 30 percent smaller than the Rayleigh wave vélocity in polycrystalline material, this technique was found to be particularly potent. Samples of 1053 carbon steel in the pearlitic state were electron beam heat treated to obtain a microstructurally modified surface layer of martensite. The Rayleigh velocity of the uniform pearlite was found to be higher by more than 2% of that of martensite. By measuring the Rayleigh wave velocity over a frequency range, thus varying the penetration depth of the Rayleigh surface waves, the modified layer depth could be nondestructively determined (fig. 15). The Rayleigh velocity remains constant as long as the Rayleigh waves probe a uniform material. When the Rayleigh waves, due to decreasing frequency or increasing wavelength, begin to sample simultaneously both the modified martensite layer and the pearlite substrate, the



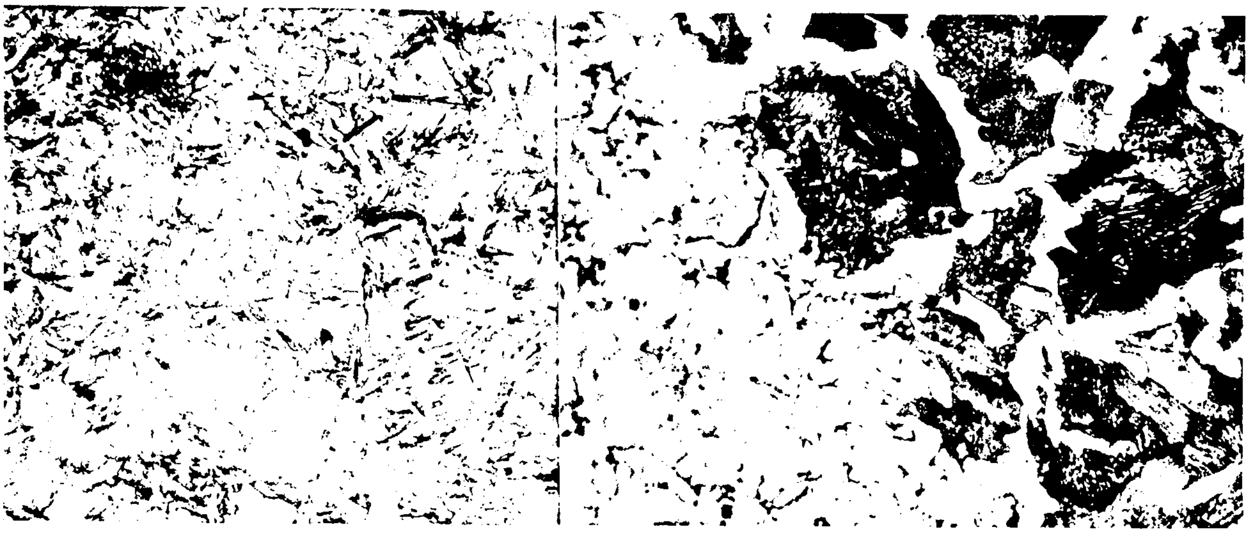
ELECTRON BEAM SURFACE MODIFICATION PROCESS

Figure 11.



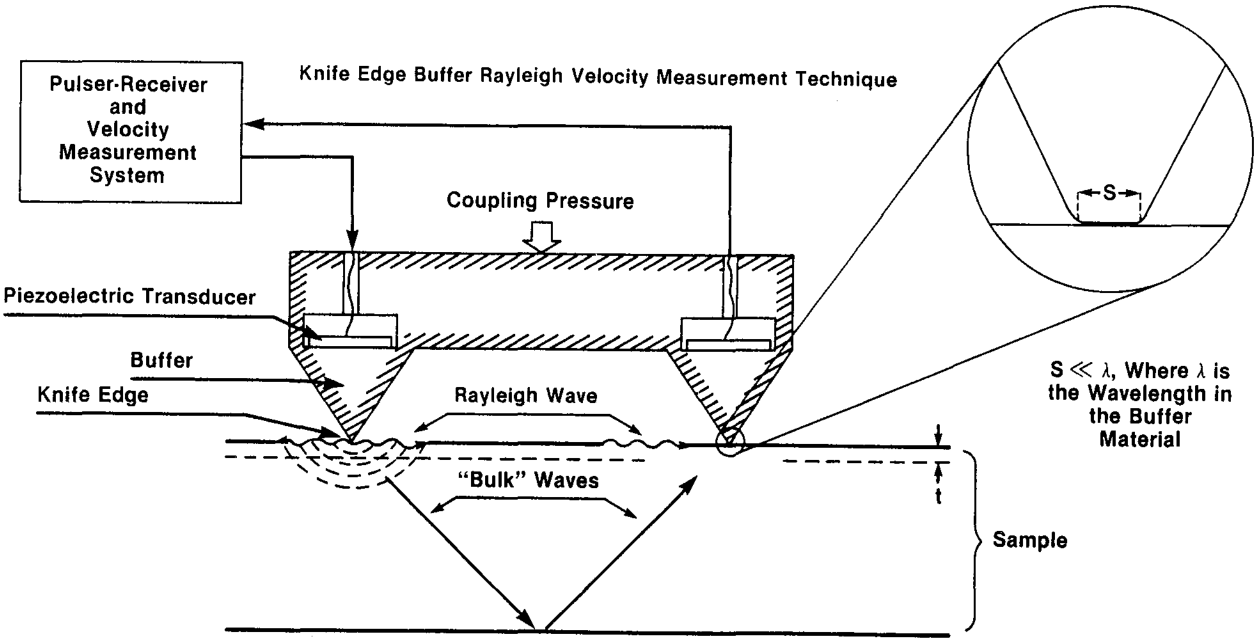
AMORPHOUS PdCuSi LAYER FORMED ON A CRYSTALLINE SUBSTRATE BY ELECTRON BEAM SURFACE TREATMENT

Figure 12.



SURFACE LAYER OF MARTENSITE (LEFT) OF AISI 1045 STEEL OBTAINED BY ELECTRON BEAM IRRADIATION OF THE PEARLITIC MICROSTRUCTURE OF THE SUBSTRATE (RIGHT)

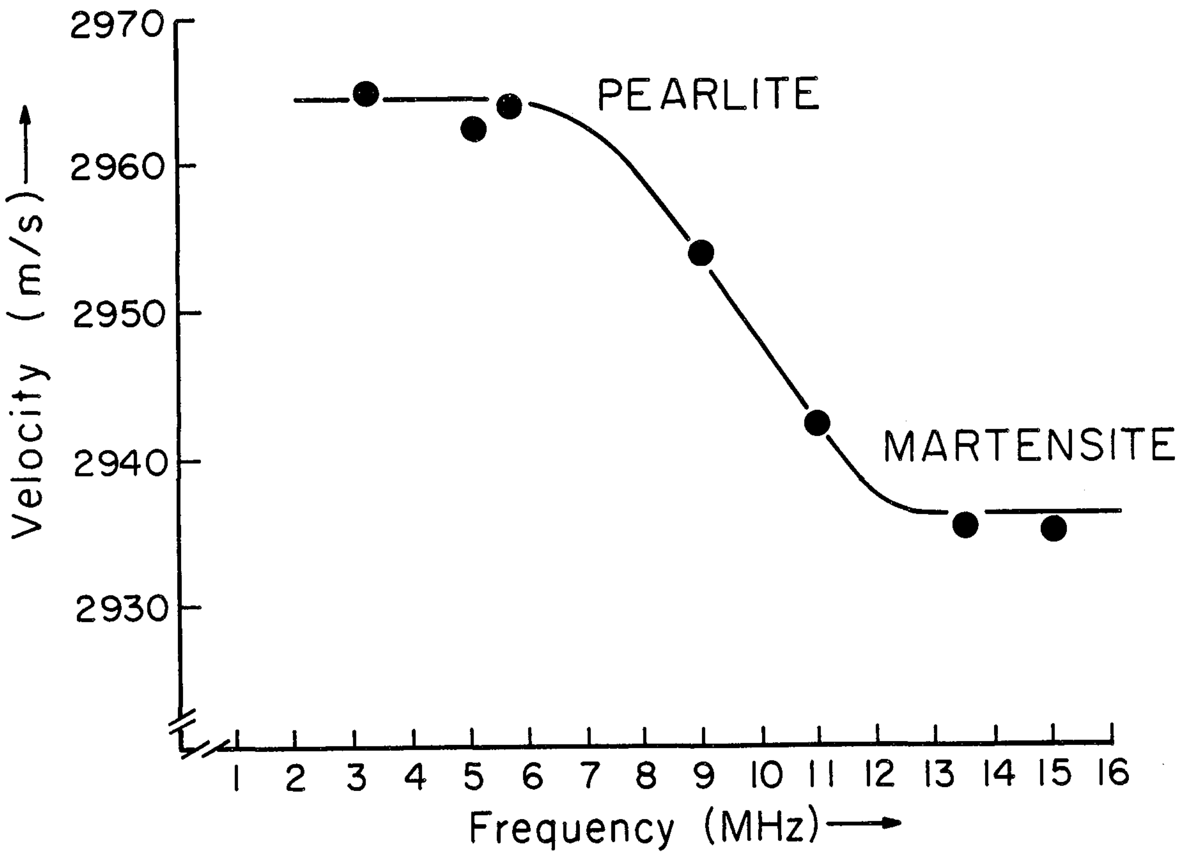
Figure 13.



- The Amplitude of the Rayleigh Wave Decreases Only Due to Attenuation Whereas the Amplitude of the Cylindrical "Bulk" Wave Decreases as $1/\sqrt{r}$, Where r is the Propagation Distance.

RAYLEIGH WAVE VELOCITY MEASUREMENTS IN MICROSTRUCTURALLY MODIFIED SURFACE LAYERS USING THE PIEZOELECTRIC GENERATION AND DETECTION BY MEANS OF KNIFE EDGE BUFFERS

Figure 14.



VARIATION OF RAYLEIGH WAVE VELOCITY WITH ULTRASONIC FREQUENCY, (RECIPROCAL OF DEPTH OF PENETRATION) IN AISI 1045 STEEL. THREE REGIONS ARE OBSERVED; a) AT HIGH FREQUENCIES (SMALL PENETRATIONS) WHERE THE VELOCITY IS CONSTANT AS LONG AS THE WAVES PROBE THE UNIFORM MARTENSITIC LAYER (ABOUT 700 μm); b) AT LOW FREQUENCIES, IN ADDITION TO THE THIN MARTENSITIC LAYER, THE PEARLITIC BULK IS PROBED; c) THE INTERMEDIATE REGION, WHERE BOTH MARTENSITE AND PEARLITE IS PROBED RESULTING IN A VARIATION OF VELOCITY, SINCE BOTH MICROSTRUCTURES ARE SAMPLED

Figure 15.

measured Rayleigh velocity is expected to increase. This behavior is apparent in figure 15. The lower curve should increase and merge asymptotically into the upper curve (straight line) that exhibits the constant Rayleigh velocity in the pearlitic substrate. These measurements demonstrate the feasibility of the technique to nondestructively characterize the properties of a modified surface layer, and to evaluate its thickness. Preliminary studies have shown that the noncontact, laser generation and laser-interferometric detection of Rayleigh surface waves can be applied for nondestructive, noncontact, evaluation of surface layers. Development of an in-process, real-time method would be of great technical importance.

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