A TECHNICAL PROPOSAL ON ULTRASONIC REFRACTORY CORROSION MEASUREMENT AT ELEVATED TEMPERATURES AND THE DEVELOPMENT OF A NONDESTRUCTIVE MEASUREMENT SYSTEM, THEREFROM*

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SUMMARY

- OBJECTIVE: To develop an ultrasonic Refractory Corrosion Measurement System (RCMS) for its applications on glass contact refractories at high temperatures.
- PRINCIPLE: Correlation of temperature-dependent ultrasonic velocity of glass contact refractory with that of its thickness/corrosion.
- FEASIBILITY: Under room temperature investigations, it has been demonstrated that measurable ultrasound can be propagated through various arrangements of refractories simulating a glass furnace.
- HIGH TEMPERATURE FEASIBILITY: By determining the velocity-dependent relationships as a function of high temperature, to measure the suitability of transducer's high temperature performance as well as to establish a criteria for accuracy and reliability.
- HIGH TEMPERATURE AND REFRACTORY-SUITABLE TRANSDUCERS: To design and manufacture ultrasonic transducers characterized by refractory-suitable acoustics, as well as their high temperature suitability.
- GLASS FURNACE EXPERIMENTS: By utilizing the velocity-dependent relationships and suitable transducers to measure the simulated refractory corrosion, while it is in contact with molten galss. Further, to compare ultrasonically measured corrosion with the actual thickness/corrosion of the start up materials.
- DEVELOPMENT OF A PROTOTYPE RCMS: By incorporating the results of this investigation design and build a Refractory Corrosion Measurement System facilitating accurate and reliable measurements at high temperatures.
- DELIVERY: Various technical reports describing the experiments, observations, and their interpretation relative to refractory corrosion measurements at elevated temperatures. Also proposed is the delivery of a prototype RCMS.
- PROJECT DURATION: Approximately one year.
- PROBABILITY OF SUCCESS: From what is known about modern ultrasound and its interaction with propagating media at room and high temperatures, it is believed that the probability of success of this proposal is between 90 to 100%. The major known risk as identified at this time is the high temperature coupling of the transducer/s. However, significant concepts have been presented in this proposal that effectively solve this problem.
- POTENTIAL BENEFITS AND THEIR OWNERSHIP/S: Besides the stipulated advantages of this proposal, it is expected that its success will generate patentable and significant business opportunities. While a critical component addressed in the proposal has been patented by Ultran Laboratories, Inc., the ownership/s of what is obtained directly through this proposal are negotiable.
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1. STATEMENT OF THE PROBLEM AND THE NEED FOR REFRACTORY CORROSION MEASUREMENT SYSTEM (RCMS)

Molten glass in a glass-making furnace is surrounded by "corrosion-resistant" refractories, which as functions of time and temperature are corroded by the chemical reactions at glass-refractory interface. Such events, if unchecked, can weaken the structure of glass furnace, and may even cause serious life-threatening and potential economic hazards.

At the present time, there are no reliable or scientific methods for the measurement of refractory corrosion, particularly at high temperatures. Glass and refractory makers use their experience and even crude methods for the estimation of refractory corrosion. Although decisions and judgements based upon such techniques are valuable, but it is not uncommon to find either premature shut-down of glass furnaces, or even catastrophic failures caused by the uncertainty of refractory corrosion. The need for a Refractory Corrosion/Thickness Measurement System (RCMS), is thus obvious.

The use of ultrasound is well-known in overt flaw detection and thickness gauging. However, until 1978, when Ultran first started, these applications were confined to the testing of metals. Commercial manufacturers of ultrasonic equipment neither understood the importance of ultrasound for the characterization of non-metals - families of ceramics and refractories - nor were they knowledgeable about the possibility of material and environment suitable acoustics and their *modus operandi*. In order to realize the reliable examination of complex-textured refractories, suitable transducers and their excitation and amplification systems must be developed or modified. This is not merely a matter of expertise in ultrasonics, but also in materials science and its applications. For the last 10 years at Ultran, we have used our knowledge of materials, studied the interaction of ultrasound with them, and developed the means for vaulable applications of ultrasound for NonDestructive Characterization (NDC). This is the basis for the development of RCMS.

OBJECTIVES

- 2.1 To determine material-suitable characteristics of ultrasonic transducer/s by using Alumina-Zirconia-Silica (AZS) fusion-cast refractory as the test material.
- 2.2 To design functional physical style of ultrasonic transducer/s, identify appropriate transducer excitation, amplification, and test system; and their implementation for the determination of right and desirable *modus operandi* at the defined temperatures.

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- 2.3 To determine the effect/s of temperature upon the velocity of longitudinal waves of AZS between 20°C (68°F) to 1500°C (2730°F).
- 2.4 To design and manufacture optimum-characteristics ultrasonic transducer/s by utilizing the data and experience from the above-mentioned objectives.
- 2.5 To develop electronic and computational mechanisms for the conversion of measured longitudinal wave velocities to the AZS distance/thickness travel, particularly as a function of thermal expansion and velocity-dependent function of temperature of AZS.
- 2.6 To apply the proto-type High Temperature Refractory Corrosion Measurement System (RCMS) to conditions that simulate AZS-molten glass contact, and to further refine the accuracy and reliability for the finalization of design, concepts, and methods.

3. PRINCIPLES OF ULTRASONIC THICKNESS MEASUREMENT

3.1 Theory of Room Temperature Measurements

By physically coupling an active ultrasonic transducer to a test material ultrasound is propagated in it. Propagation of ultrasound will be interrupted when the traveling wave encounters a physical discontinuity or gross chemical heterogeneity. In a continuous medium this condition is represented by the far side of the test material, which acts as a reflector of the wave. For details on the principles and methods of ultrasonic characterization, see Bhardwaj (1986).¹

Velocity/thickness measurement devices are based upon the interpretation of measured time-of-flight with known material travel distance or its velocity. For example, if the time-of-flight of ultrasound travel is t, and the materials distance traveled is D, then the velocity of sound

$$V = D/t$$

When the examination is conducted from one side of the test material, such as in thickness gauging by a single transducer (acting simultaneously as transmitter and receiver), or by two transducers placed side-by-side (one acting as a transmitter and the other as a receiver of ultrasound), the material distance is traveled two times. Therefore, the measured time-of-flight corresponds to the round-trip distance in the test material, or

$$V = 2D/t$$

In order to determine an unknown thickness by ultrasound, velocity of sound through it must be known. All thickness gauging methods utilize this principle and the clock of a gauging pulser-receiver consists of a mechanism that takes of account the round-trip times-of-flight, as follows:

$$D = V \times t/2$$

Based upon this principle, thicknesses can be directly measured from realtime rf A-scans or by digital thickness gauges. Depending upon the test material composition and texture and the objectives of testing, a number of transducer coupling methods can be used. These are:

a. Single direct contact transducers,

4

- b. Single delayed contact transducers, and
- c. Dual T-R transducers separated by short delays.

Example of an instrument that facilitates such measurements by single direct contact method is Ultran's Model 4551 Thickness/Velocity Meter. It should be noted such instruments are designed in a manner that their operation is relatively simple. Furthermore, they are normally used for denser materials testing and do not have provision for the analysis of coarse-grained materials, particularly at higher temperatures.

3.2 Theory of High Temperature Measurements

As shown in the preceding section, it is a relatively easy to measure thickness/corrosion of a material if the test is carried out under normal conditions. In the present case the conditions of testing are compounded by the fact that the test materials are not only at relatively high temperatures, but that they are also under varying thermal gradients as well as characterized by coarse-grained textures. Therefore, we must establish a procedure that would facilitate the determination of velocity of ultrasound through test materials at specified thermal conditions. Consider, for example,

 D_0 = Room temperature thickness of the sample

T = temperature of measurement

DT = Thickness of the sample at temperature T

to = Time-of-Flight of ultrasound through thickness Do

 $t_T = TOF$ at temperature, T, then

 V_0 (velocity of ultrasound at room temperature) = D_0/t_0

 V_T (velocity of ultrasound at temperature, T) = D_T/t_T

DT, thickness of the sample as a function of temperature is given by,

 $D_0 + \Delta D_T$, where

ΔD_T is the linear thermal expansion of the sample at temperature, T. Thus, velocity as a function of temperature can be completely defined as,

$$V_{T} = (D_{0} + \Delta D_{T})/t_{T}.$$

If velocities, V1,V2,V3,,,,Vn are measured at T1, T2, T3,,,,Tn temperatures on a known thickness, D of refractory block, then a "linear velocity contraction co-efficient" relationship analogous to "linear thermal expansion Co-efficient," can also be established, such as

$$\Delta V_{T}(1,2,3,n) = f[\Delta D_{T}(1,2,3,n)].$$
 8

Whereas, for a majority of solids the linear thermal expansion has a positive value, for "linear velocity contraction," a negative number is more logical. Hypothetical relationships describing equations 7 and 8 are shown in Figures 1 and 2.

The practical significance of relationships established by equations 7 and 8 is realized when it is desired to ultrasonically measure the thickness of a refractory, particularly

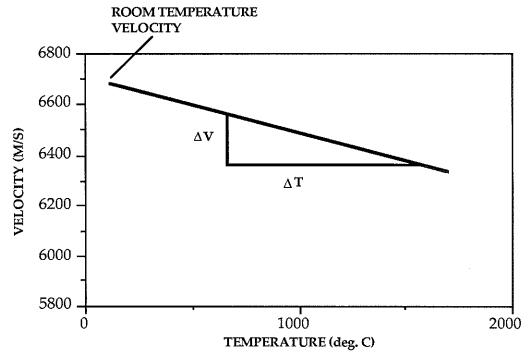


Fig. 1. A hypothetical relationship between ultrasonic velocity as a function of temperature, also defining the slope,

 $\Delta V/\Delta T$.

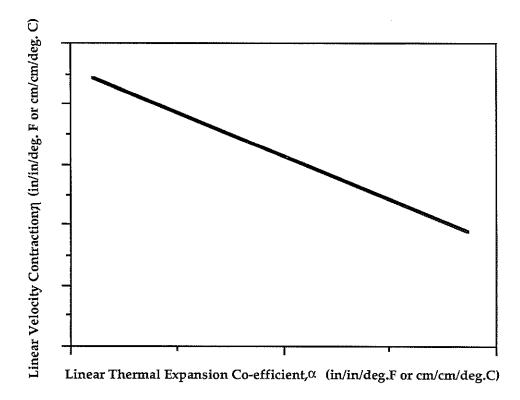


Fig. 2. A hypothetical relationship between Linear Thermal Expansion Co-efficient and Linear Velocity Contraction Co-efficient.

when it is subjected to a thermal gradient. Such situations occur realistically in the field applications of refractories. Consider, for example a refractory block of unknown thickness, Dx, subjected to a thermal gradient of 200°C to 1500°C as shown in Fig. 3, and it is desired to determine its thickness by ultrasonic method. However, before this can be accomplished, a velocity value corresponding to the specified thermal gradient needs to be determined. This is given by

$$V_{T'} = V_0 + \Delta V_{T'}$$

Where $V_{T'}$ is the true velocity of the material at a specified thermal gradient, and $\Delta V_{T'}$ is the velocity contraction factor at the specified thermal gradient. This is accomplished from velocity and temperature relations, Fig. 1. From this relationship determine the value of $\Delta V_{T'}$ by subtracting the velocity at 200°C from one at 1500°C. This is shown in Fig. 4. If the velocity of AZS at room temperature is 6700m/s and the measured ΔV (from Fig. 2) is 290m/s then,

$$VT'(200^{\circ}C \text{ to } 1500^{\circ}C) = 6700 + (-290) = 6410 \text{m/s}.$$
 10

The negative value for $\Delta V_{T'}$ indicates that ultrasonic velocity actually decreases as the temperature increases.

Since, the $t_{T^{\prime}}$, the time-of-flight through the material under a thermal gradient, is a real measurement, then

$$Dx = 641,000 \times tT' \text{ cm.}$$

If, however, the extreme precision is not needed to determine the thickness of a refractory as a function of thermal gradient, then one could use a $V_{T'}$ value at a specific temperature from Fig. 1. It is 6380 m/s at 1500°C . If all other factors were constant, then the difference in the values of Dx measured by eq. 11 and by the "lesser" accurate method just cited is, approximately 0.5%.

Experimental relationships describing equations 7, 8, and 9 will reveal the following:

- 1. Unkown thicknesses of refractory at elevated temperatures.
- 2. Empirical relationship between ultrasonically determined thickness values with the known ones.
- 3. Application of this knowledge for the development of an "ergonomic" RCMS, which incorporates the effects of temperature upon the velocity of ultrasound in order to enhance the accuracy and reliability of the proposed method.

4. BACKGROUND RELATED TO REFRACTORIES

Success of any diagnostic application of ultrasound depends upon the quality of signals, representative of propagation of ultrasound through a given set of materials under specified environmental conditions. In this section we show several conditions representing the relationships of refractories, molten glass, and transducers.

4.1 Conditions under which ultrasound propagation is feasible

4.1.1 When the transducer is in direct contact with AZS: When the path of a propagating ultrasonic wave is uninterrupted, i.e., when it goes through a

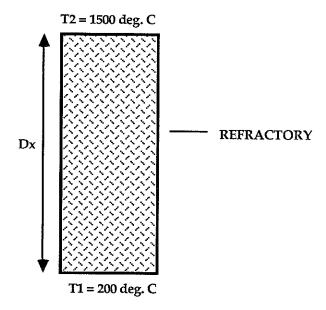


Fig. 3. A refractory block of unknown thickness Dx low temperature side at T1 = 200 deg. C, and the high temperature side at T2 = 1500 deg. C.

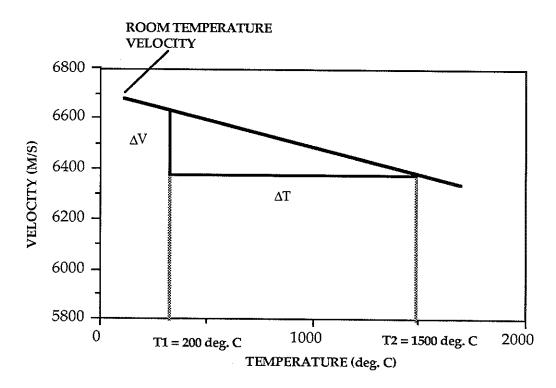


Fig. 4. Measurement of temperature effect upon velocity - $\Delta\!\!I$ - from 200 to 1500 deg. C.

continuous medium, it travels "most" freely. Such a condition simulating a glass melter is shown in Fig. 5. Here the transducer is in direct contact with the AZS block, which on its other side supports molten glass. It is reasonable to imagine that the appropriate ultrasonic wave will be reflected at the AZS-glass interface to the transducer.

4.1.2 When the transducer and AZS are separated by cemented or liquid-filled interfaces: It is not uncommon to find AZS or other fused-cast glass melter refractories being protected by insulating refractories. If reasonable ultrasound is expected to propagate through such a condition, it is imperative that the interface between AZS and insulating refractory be either cemented, or filled with glass; the latter condition being more preferable. A schematic representation of this condition is shown in Fig. 6. Such an arrangement of refractories will cause a degree of ultrasonic attenuation at the insulating material - AZS interface, thus allowing lesser energy propagation to the AZS-glass interface. Ideally, it is desirable for the transducer tobe in direct contact with AZS refractory. However, we will show that ultrasound can be propagated and further optimized even under complex arrangement of refractories.

4.2 Conditions under which ultrasound propagation is not feasible

Should there exist an empty space or an air gap in the path of ultrasound it "will not" propagate through it. Such a condition would exist if an AZS block was simply "resting" on an insulating refractory, i.e., this interface was neither cemented, nor filled by glass. This condition is shown in Fig. 7 and if it exists at a given point, then it would be virtually impossible for ultrasound to make its way up to AZS-glass interface. This ultrasonic pheomenon is best understood when the propagation of ultrasound is viewed as a boundary layer problem.

Transmission of ultrasonic wave from one medium to another is determined by the efficiency with which it propagates through the interface between the media. This is best described by acoustic impedance - product of velocity of sound and density - of the materials involved, such as:

$$T = 4Z_2Z_1 / (Z_2 + Z_1)^2$$

Where T = Transmission co-efficient, $Z_1 = Acoustic$ impedance of the medium of transmission, i.e., one in contact with transducer, and $Z_2 = Acoustic$ impedance of the medium of propagation. Similarly,

$$R = [(Z_2 - Z_1) / (Z_2 + Z_1)]^2$$
13

Where R = Reflection co-efficient, and other terms having usual meanings.

Now consider for example propagation of ultrasound from a fused silica insulating refractory to AZS. First, we must contend with a "finite layer of air" at this interface. No matter how small it may be, the fact is that it is a medium of propagation and we must solve for transmission and reflection co-efficients by accounting for the acoustic impedances of fused silica and air, yielding:

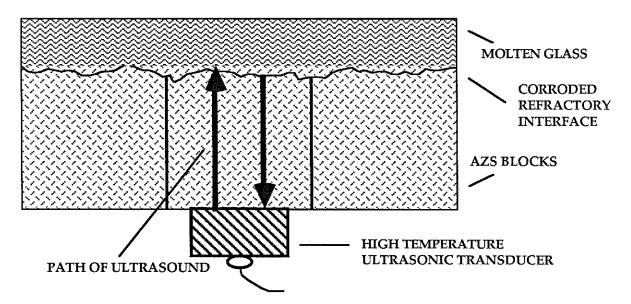


FIG. 5. Relationship of refractory, molten glass, and transducer facilitating the propagation of ultrasound to refractory-glass interface - when the same refractory block is in direct contact with transducer and molten glass.

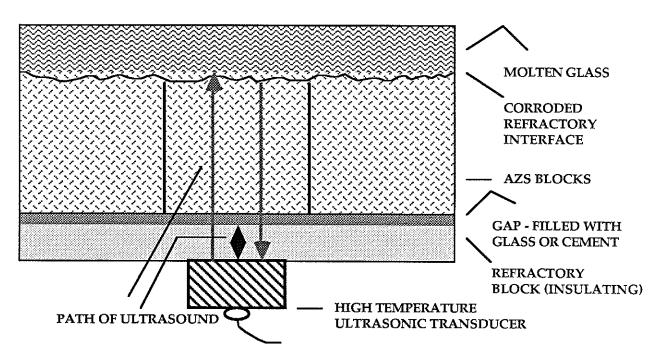


FIG. 6. Relationship of refractory, molten glass, and transducer facilitating the propagation of ultrasound to refractory-glass interface - when several refractories and other interfaces are involved. In the case shown here, ultrasound is propagating from a material, such as an insulating refractory, to a gap filled either by glass, or by a cement to AZS and finally to the AZS-molten glass interface.

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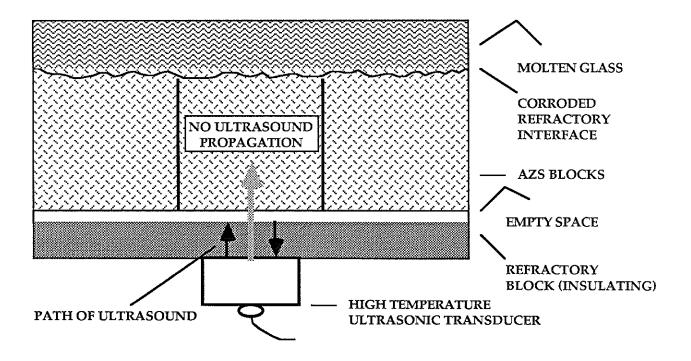


FIG. 7. Relationship of refractory, molten glass, and transducer impeding the propagation of ultrasound to refractory-glass interface. In the case shown here, ultrasound travels into a material, such as an insulating block, from where it is reflected right back to the transducer due to empty or air-filled gap between insulating block and AZS,

R = 99.9999

These observations indicate clearly that virtually all ultrasonic energy is reflected from fused silica-air interface, thus allowing no propagation into AZS block, the material of interest. A similar reasoning will apply if the propagating ultrasound encountered gross defects in AZS, such as large casting voids.

5. SMALL SCALE ROOM TEMPERATURE SIMULATIONS

In order to exhibit the principles and methods of ultrasonic examination of AZS-glass interface, several observations were made on a variety of combinations of refractories. Described in this section, these observations were carried out at bench-scale and at room temperature. Besides the controlled feasibility, these observations are meant to support the arguments presented in section #4. Materials used in this simulation are real, but not necessarily representing the dimensions expected in the field environment.

5.1 When transducers are in direct contact with AZS

A 0.50in thick glass sheet was placed on a 5.0in section of AZS block with a small amount of glycerene in between, simulating the condition of wettability of AZS by molten glass. Ultrasound from 1.0MHz transducer - in direct contact with AZS - was propagated by direct reflection method, Fig. 8. The real time oscilloscope trace of this setup is shown in Fig. 9. Indications corresponding to the thicknesses of AZS and glass are identified in the captions of this figure.

5.2 When transducers are in indirect contact with AZS

This is an extension of the setup in section #5.1, with the exception that a coarse-grained silica refractory block is inserted between the transducer and AZS. A thin sheet of glass - 0.035in - was placed with glycerine on both sides between AZS and silica refractory, simulating a condition of glass penetrating at the silica refractory and AZS interface. Details of this arrangement are shown in Fig. 10. The realtime oscilloscope trace of this setup is shown in Fig. 11. Note that indication from silica-AZS (thin glass sheet) interface #2 is much larger than the one from the top of AZS-glass interface. For further explanation, see section 4.1.2.

5.3 When air gap exists between refractory arrangements

In order to simulate the condition described in section #4.2, the AZS block was placed on a silica refractory without any liquid at the interface, Fig. 12. The time domain trace of this setup, Fig. 13, shows that ultrasound traveled only within the silica refractory with no measureable propagation into AZS, the material of interest.

6. ACOUSTICS SUITABLE FOR REFRACTORIES MEASUREMENTS

While there is no doubt about the fact that ultrasound is capable of providing more significant information about the media through which it travels than mere metals-NDT-type-flaw-detection. The other fact - generally given lesser importance - is that the test-material-suitable-acoustics be identified, particularly when considering the examination of highly complex and heterogeneous materials such as refractories.

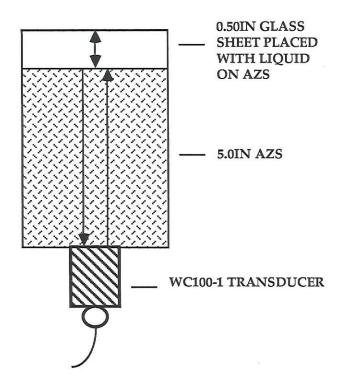


FIG. 8. Simulation of refractory glass contact when transducer is directly placed on AZS surface.

Reflection coefficient at AZS-glass interface, R = 7.4%

It is sufficient to reflect ultrasound from this interface, see the figure below

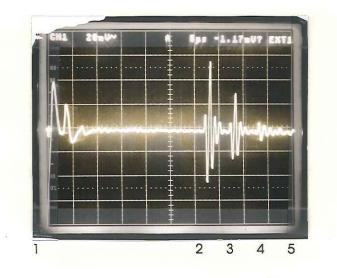


FIG. 9. A realtime oscilloscope trace showing the ultrasonic response from the set up in Fig. 5.

#1: Initial pulse

#2. Interface between AZS and

glass sheet

#3. Interface between glass and air

#4, 5, ... Multiples of glass thickness.

Time-of-flight from 1 to 2: corresponds to the thickness of AZS.

Time-of-flight from 2 to 3: corresponds to the thickness of glass sheet.

Frequency used for demonstration: 1.0MHz with >50% bandwidth.

Method: Direct Reflection.

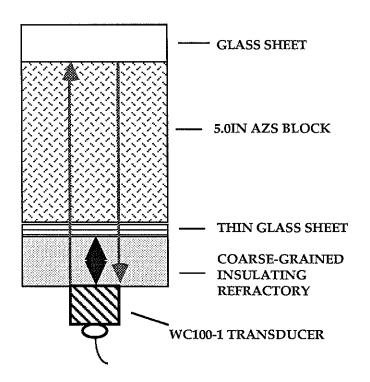


FIG. 10. simulation of refractory glass contact when the transducer is placed indirectly on the refractory. In the example shown AZS is separated by a coarse-grained fused silica, the interface of which is filled with glass.

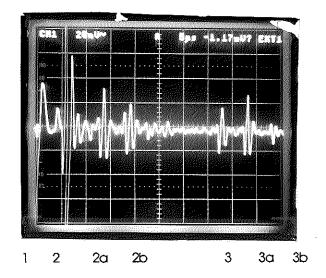


FIG. 11. A realtime oscilloscope trace showing the ultrasonic response from the set up in Fig. 10.

#1. Initial pulse#2. Interface between coarse-grained refractory and AZS#s2a, 2b .. multiples.#3. Interface between AZS and glass sheet.

Time-of-flight from 1 to 2: corresponds to the thickness of coarse-grained refractory.

Time-of-flight from 2 to 3: corresponds the thickness of AZS.

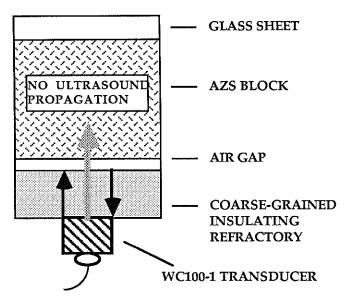


FIG. 12. Simulation of refractory glass contact when the transducer is separated from the refractory by an air gap, such as the one produced when two materials are in contact without cement, liquid, or glass.

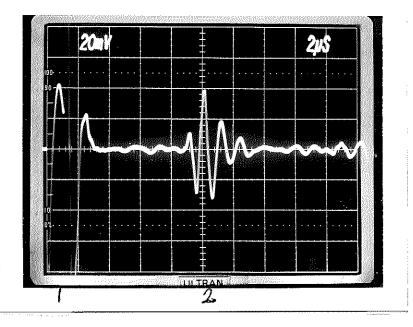


FIG. 13. A realtime oscilloscope trace showing the ultrasonic response from the set up in Fig. 12.

#1. Initial pulse

#2. Interface between coarse-grained refractory and air gap with AZS.

Time-of-flight from 1 to 2: corresponds to the thickness of coarse-grained refractory block.

Frequency used for demonstration: 1.0MHz with >50% bandwidth.

Method: Direct Reflection.

6.1 Ultrasonic nature of refractories

Refractories not only vary in chemical compositions, but they vary more dramatically with respect to textures. Take for example, an isostatically-pressed zirconia or alumina (nearly 100% dense) to fused silica grain - between 20 to 40% porous! From the standpoint of suitable characteristics of ultrasound, the former may require 2 to 15MHz frequencies, while the latter, from 50KHz to 0.5MHz. Ultrasonic characteristics are more sensitive to textural variations than anything else while working with ceramics. These observations and their significance have been recently described in an attempt to present a more scientific picture of NDC, Bhardwaj (1987).^{2,3}

6.2 Importance of short pulse VLF transducers

Confined to the objectives of this proposal, we assume that the refractory materials of interest are attenuative, coarse-grained, large crystal size materials of granular to inter-locking textures. Not only are such materials great scatterers of ordinary ultrasound (generally, >1.0MHz), but they are also characterized by relatively low ultrasonic velocities from approximately 2000 to 4000m/s. Scattering phenomenon will limit the higher frequency usage, while lower velocities (or shorter wavelengths at specified frequencies), particularly at less than MHz frequencies, will tend to increase the size of aperture, i.e., the active area of transducer. These factors complicate the proper and desired examination of coarse-grained refractories. The subject of transducer dimensions will be described in the physical transducer design section; here we outline significant advances in Very Low Frequency (VLF) ultrasonics of particular importance to this proposal.

It is the experience of this investigator that in order to characterize refractories for corrosion or for microstructure, frequencies beyond 1.0MHz will have only limited value. Attenuation and scattering are the predominant reasons for this limitation. From our examination of a number of refractories, we conclude that suitable frequencies are generally from <100KHz to <1.0MHz. Indeed, at first glance it appears that the resolution would be minimized due to reduced frequency. We should like to introduce the fact that resolution has more practical meaning when it is viewed in light of a transducer pulse width rather than its frequency, Bhardwaj (1987)² and Ultran (1987).⁴ The point is that if we shorten the pulse width of a VLF transducer, then we achieve optimum resolution and optimum strength of ultrasonic energy in order to propagate through attenuative refractories.

These observations were made by this author nearly 15 years ago. However, at that time neither the subject of complex materials characterization by ultrasound was properly understood, nor was there transducer technology that would produce 2 or 3 λ VLF transducers. Imagine a 250KHz device with a pulse width of 10 miliseconds, and a 150KHz of 30 miliseconds! These undesirable VLF acoustics prevented realistic, accurate, and reliable applications on refractories - pointing out that the transducer technology had to be developed further. Our extensive R & D into ultrasonic technology and its NDC applications during the last 10 years have resulted in the generation of even very short pulse width VLF transducers, as well as a comprehensive range from <100KHz to >100MHz, Ultran (1986). As a historical reference, the need for such developments in ultrasound was directly responsible for the inception of Ultran.

6.3 State of the art VLF technology

In order to establish a workable terminology, we regard all ultrasonic transducers from 50KHz to 500KHz as falling in the under Very Low Frequency (VLF) category. Due to the large capacitive loads, VLF transducers must an rf burst that facilitates pulse width control, besides high energy required to drive VLF transducers. These features are present in Ultran's high energy HE-900 system. This system is now beyond prototypical stage.

Fig. 14 is a realtime rf oscilloscope trace demonstrating the propagation of short pulse 250KHz ultrasonic energy into an 8.0in fused silica grain refractory, presumably used as crown brick in the glass furnaces. Although AZS is lesser attenuative than this material, it shows that "good" quality ultrasound can be propagated even through highly attenuative media. Good signal quality is important for reliable and accurate measurements of time-of-flight or velocities, as well as for higher resolutions of thinner sections of material travel.

Fig. 15 is a composite of time and frequency domain analysis of a 50KHz-short-pulse-width transducer. Its measured pulse width is only 2.5λ or 50ms, while the bandwidth is 60% of the center frequency.

Fig. 16 is another example of a VLF transducer of 100KHz frequency. Its measured pulse width is 2.0λ or 20ms, and bandwidth is nearly 70% of the center frequency.

Fig. 17 is a similar example of a 250KHz transducer. Its pulse width is 2.5λ or 10ms, and bandwidth is 60% of the center frequency.

In simple terms, any reflecting target including the resolution of distance traveled by ultrasound can be measured after the width of ultrasonic pulse. For example, a 250KHz short pulse transducer is capable of resolving approximately 2.0in of AZS. Further moredue to higher VLF energy it should be capable also of longer material travel, also.

Fig. 18 is a time and frequency domain analysis of a "long" pulse width 250KHz transducer. Its measured pulse width is approximately 7.0λ or 30ms, and 25% bandwidth. While longer pulse width transducers are not desirable for higher resolutions, they have other advantages. Energy of longer pulse width transducers is much higher than those of their short pulse width counterparts. Furthermore, such devices can be extremely useful as receivers, particularly when extreme optimization of ultrasonic parameters are needed in a given application. We shall describe these factors in details while considering the parameters for optimum RCMS transducer designs.

In this section we have demonstrated the successful development of VLF transducers for their applications on attenuative media and longer material travels. A variety of other transducers are described elsewhere, Ultran (1986).⁵

7. DEVELOPMENT OF ACOUSTO-GEOMETRICAL PARAMETERS

In order to achieve the desired NDC objectives of any given application of ultrasound, such as for thickness measurement, micro-structure evaluation, or property correlations, the first and the foremost task is to identify and develop material-suitable acoustic and geometrical parameters of ultrasound. This is the task of a transducer designer who also must understand the physical nature of ultrasound when it interacts with a given material. In a nutshell, this is the preamble of Ultran's practical approach to the solution of a given problem via ultrasound. In this section we describe the step-wise approach

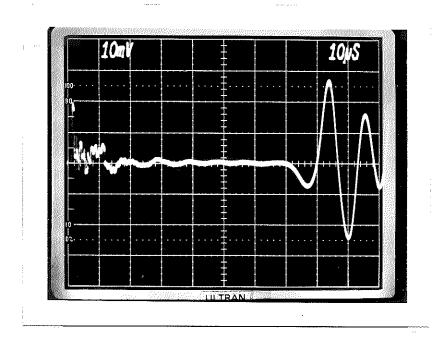


FIG. 14. A realtime oscilloscope trace showing the propagation of short pulse width 250KHz ultrasound through a highly attenuative, porous, and coarse-grained fused silica refractory.

Thickness investigated: 8.0in

Velocity of sound (measured): 115,000in/s

Method: Direct Transmission.

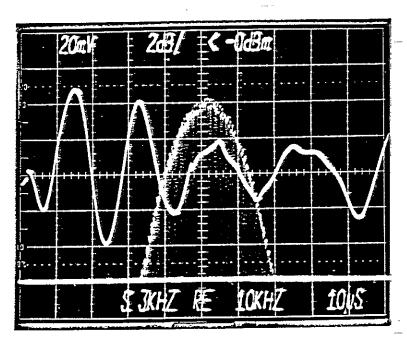


FIG. 15. Time and frequency domain analysis of a Very Low Frequency (VLF), but short pulse ultrasonic transducer.

Time Domain V.S. = 20mV/DH.S. = 10 microsec/D

Frequency Domain V.S. = 2dB/DH.S. = 10KHz/D

Example shown: ULTRAN - WS100-0.05

Nominal Frequency: 50KHz Active area diameter: 1.0in Acoustic Series: ULTRAN "W"

Measured Parameters: Center Frequency: 50KHz Bandwidth at -6dB: 60% of CF

Pulse Width: Approx. 2.5 wavelengths or

50 microsec.

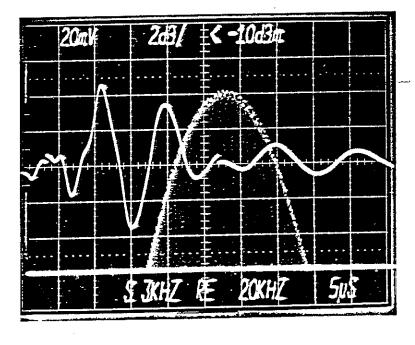


FIG. 16. Time and frequency domain analysis of a Very Low Frequency (VLF), but short pulse ultrasonic transducer.

Time Domain V.S. = 20mV/D H.S. = 5 microsec/D

Frequency Domain V.S. = 2dB/DH.S. = 20KHz/D

Example shown: ULTRAN - WS100-0.1

Nominal Frequency: 100KHz Active area diameter: 1.0in Acoustic Series: ULTRAN "W"

Measured Parameters: Center Frequency: 110KHz Bandwidth at -6dB: 66% of CF

Pulse Width: Approx. 2.0 wavelengths or

20 microsec.

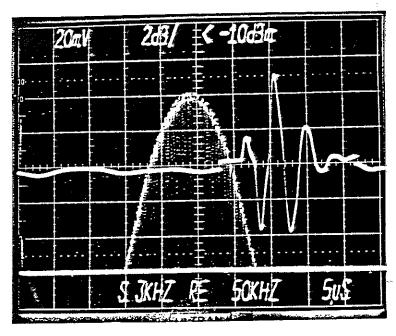


FIG. 17. Time and frequency domain analysis of a Very Low Frequency (VLF), but short pulse ultrasonic transducer.

Time Domain V.S. = 20mV/D H.S. = 5 microsec/D

Frequency Domain V.S. = 2dB/D H.S. = 50KHz/D

Example shown: ULTRAN - WS100-0.25

Nominal Frequency: 250KHz Active area diameter: 1.0in Acoustic Series: ULTRAN "W"

Measured Parameters: Center Frequency: 240KHz Bandwidth at -6dB: 60% of CF

Pulse Width: Approx. 2.5 wavelengths or

10 microsec.

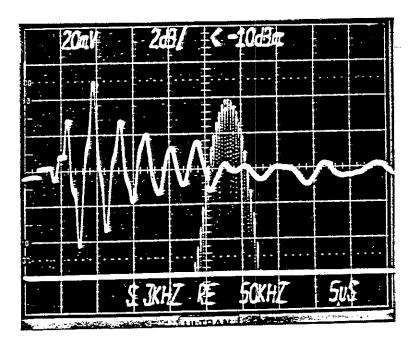


FIG. 18. Time and frequency domain analysis of a Very Low Frequency (VLF), but long pulse ultrasonic transducer.

Time Domain V.S. = 20mV/D H.S. = 5 microsec/D

Frequency Domain V.S. = 2dB/D H.S. = 50KHz/D

Example shown: ULTRAN - KS100-0.25

Nominal Frequency: 50KHz Active area diameter: 1.0in Acoustic Series: ULTRAN "K"

Measured Parameters: Center Frequency: 270KHz Bandwidth at -6dB: 25% of CF

Pulse Width: Approx. 7 wavelengths or

30 microsec.

towards the development of "right" acoustic and geometrical parameters by taking AZS as an example material.

7.1 Development of AZS suitable Acoustics

The most workable definition of optimum ultrasonic frequency for a given material is the "highest" frequency acceptable by the material that exhibits "minimum" attenuation. Applications of frequencies in excess of the optimum would only result in their attenuation, thus reduction of the intensity of reflected or transmitted ultrasonic signals. This phenomenon and its importance in materials characterization is defined in a short note, "Optimum Ultrasonic Parameters for Low Density Composite Evaluation," Bhardwaj (1983).⁶ Although this work was demonstrated on fiber-reinforced-plastic and C-C composites, its principles will be applied to AZS frequency optimization.

In order to develop AZS-suitable acoustics, it is proposed that several samples of AZS, representing its skin and core be analyzed from 250KHz to 2MHz frequencies over a reasonable thickness range of the material. Such an analysis will define the following critical ultrasonic parameters suitable for AZS measurement:

- a. Optimum ultrasonic frequency,
- b. Optimum pulse shape and size,
- c. Resolution limits,
- d. Frequency-limited scattering, and
- e. Design parameters for receiving transducers.

To quantify this information is the objective of this section. However, some general conclusions can be drawn here. Assuming 6000m/s as the longitudinal wave velocity for AZS, we can realistically develop ideas about resolution, pulse size, and scattering. These observations are shown in Table I.

FREQUENCY	WAVELENGTH ¹ λ mm	THEO.RES. ² λ/2 mm	REALISTIC ³ PULSE m m	REALISTIC RES. ⁴ m m	SCATTER ⁵ BEYOND CRYSTAL Ф mm
100KHz	60	30	180 ^a	180	60
250KHz	24	12	60 ^b	60	24
500KHz	12	6	24 ^a	24	12
1.0MHz	6	3	₁₂ d	12	6
2.0	3	1.5	4.5 ^e	4.5	3
3.0	2	1	3f	3	2

TABLE I. Wavelength, resolution, and scatter relationships as a function of various ultrasonic frequencies determined by assuming longitudinal wave velocity of 6000m/s for AZS.

Explanation of terms in Table I.

1. $\lambda = v/f$; 2. Not realistic to expect theoretical resolution with this material; 3. These are real numbers achievable at specified frequencies, for example: $a = 3\lambda$; $b = 2.5\lambda$; c =

 2.0λ ; $d=2.0\lambda$; and $f=1.5\lambda$. 4. It is assumed that resolution of reflecting targets will begin "immediately" after the time occupied by the pulse in time domain; 5. Scattering of ultrasound is generally assumed to begin when wavelength and scatterers (grain size, porosity, grain boundaries, etc.) are within the proximity of each other. For further explanation of #s 3 and 4, see section #6.

Concluding from the observations in Table I, it is feasible to achieve the stipulated thickness range from 12mm (0.5in) to 365mm (14.0in) measurement in AZS. However, it may not be possible to do so with a single transducer. Resolution and accuracy of measurement are dependent upon each other, and should be examined as functions of scattering. This should be done for two reasons: first, AZS is highly crystalline, and secondly, corrosion at AZS-molten glass interface will introduce scatter at certain frequency. For example, while a 1MHz frequency would resolve 12mm or less in AZS, scattering caused by crystal sizes of approximately 6mm may obliterate the accuracy of thickness measurements and the attenuation of the signal. On the other hand, higher frequency is likely to yield more precision at the AZS-molten glass interface, provided the interface does not introduce scatter at a given higher frequency. A lower frequency may not facilitate higher resolution, but it would travel much longer distances and yield corrosion measurements, corresponding to "some" integrated value of corrosion roughness. In short, we must consider average crystal size of AZS and some quantitative description of corrosion at AZS-glass interface as functions of interrogating frequency and its pulse shape and size in order to arrive at reasonable definitions for resolutions, accuracies, and precision. These are the objectives of this section.

7.2 Development of AZS suitable geometry of ultrasound

Once the suitable frequencies have been determined for AZS, the next proposed step is to determine the shape and size of the active areas of ultrasonic transducers. While doing this we must notice that it is desirable to make measurements in AZS from 12mm (0.5in) to 365mm (14.0in). From the practical standpoint it may be desirable to have a "smallest" transducer, but realistically, the transducer size should be dictated by the selected frequency and expected travel distance of ultrasound. Ultrasound is not characterized by rectilinear propagation even in the near or Fresnel zone, and beyond this zone, it (ultrasound) suffers "extensive" diffraction. These effects are described in details elsewhere, Bhardwaj (1986). Effects of convergence and divergence of ultrasound become more severe when considering relatively lower frequencies and ultrasonic velocities. Briefly, these relations are described below:

Fresnel Zone =
$$D^2/4\lambda$$
 16

where D = dimensions (generally, diameter) of the transducer, and λ = wavelength of ultrasound. The region beyond the Fresnel zone is known as the far or Fraunhofer zone, and

their point of contact as the transitional zone. At this transition point the propagating ultrasound experiences diffraction, defined as half this angle by,

$$\theta = \sin^{-1.22} \lambda / D$$
 17

Ideally, it is desirrable to conduct ultrasonic measurements at or around the nf/ff zone and at minimum diffraction or divergence of propagating ultrasound. However, these desires are invariably compromised. From our experience, we find that the "best" working range of a transducer is

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The conditions that would describe the optimum geometry of ultrasound for AZS measurements can be best described by assuming either certain dimensions of the transducer, or by the material travel distance. Further, this exercise also must take a note of the selected frequencies and velocity of longitudinal waves in AZS, eq. 15 and 16. Tables II and III are such computations showing the extents of nf/ff and diffraction as functions of frequencies, and assumed active area diameters of several transducers for AZS. The purpose of these tables is to determine suitable transducer dimensions at specified frequencies. The reason for assuming circular transducers is strictly for simplicity. We are not concluding the shape or size for the final transducer design. However, this table should serve as an excellent guide in making these decisions.

TRANSDUCER ACTIVE Φ (in)		FREQUENCIES nf/ff ZONES IN in			
	<u>100KHz</u>	250KHz	<u>500KHz</u>	<u>1.0MHz</u>	<u>2.0MHz</u>
0.5	0.03	0.065	0.13	0.26	0.52
1.0	0.106	0.26	0.526	1.05	2.10
1.5	0.24	0.57	1.18	2.37	4.8
2.0	0.425	1.05	2.10	4.2	8.3
2.5	0.664	1.64	3.3	6.6	13.3
3.0	0.96	2.4	4.7	9.5	19.1
3.5	1.3	3.22	6.45	12.9	26.0
4.0	1.70	4.21	8.42	16.8	34.0
4.5	2.15	5.32	10.65	21.3	43.0
5.0	2.65	6.6	13.15	26.3	53.2

TABLE II. nf/ff zones in AZS refractory as functions of transducer frequencies and dimensions. Velocity of longitudinal waves in AZS = 6000m/s (236,000in/s).

TRANSDUCER ACTIVE Φ (in)			FREQUENCIES (HALF DIFFRACTION ANGLES ⁰)			
	<u>100KHz</u>	250KHz	500KHz	<u>1.0MHz</u>	2.0MHz	
0.5				35.8	10.9	
1.0			34.9	17.0	5.5	
1.5		49.8	22.5	11.25	3.6	
2.0		35.0	16.7	8.4	2.7	
2.5		27.3	13.2	6.7	2.2	
3.0	<i>7</i> 3. <i>7</i>	22.5	11.0	5.6	1.8	
3.5	55.3	19.1	9.4	4.8	1.6	
4.0	46.0	16.7	8.2	4.2	1.4	
4.5	39.8	14.7	7.3	3.7	1.2	
5.0	35.2	13.3	6.6	3.4	1.1	

TABLE III. Half diffraction angles in AZS refractory as functions of transducer frequencies and dimensions. Velocity of longitudinal waves in AZS = 6000m/s (236,000in/s).

Data in Tables II and III serves an excellent guide for the selection of suitable transducer dimensions in order to interrogate AZS from 0.5 to 14.0in once optimum frequencies for the material have been determined, section #7.1. While these tables provide a mechanism for the design of "ideal" transducers, it should be noted that there are limitations to what can be practically achieved. The current piezoelectric manufacuring technology limits the maximum dimensions to 3 to 3.5in diameters for the transducers required under this proposal for high temperature use. While creating an appropriate transducer design, these factors will be given due importance along with various other factors discussed thus far.

8. PHYSICAL TRANSDUCER DESIGN

After making the decision relative to suitable transducer frequency and dimensions, transducers will be packaged to meet the requirements of AZS measurement at high temperatures. This section describes all potentially applicable physical styles along with their high temperature counterparts.

8.1 Basic suitable physical styles

There are three basic physical styles of transducers that are used in materials testing when access to its surfaces is only from one side. These are:

- 8.1.1 Single transducer in direct contact with refractory. See page 9 of Ultran Publication U-777.
- 8.1.2. Single transducer in contact with refractory through a delayed buffer rod. See page 13 of Ultran Publication U-777.
- 8.1.3 Dual T-R transducers in contact with refractory though small specifically angulated buffer zones. See page 11 of Ultran Publication U-777.

8.2 High temperature suitablity

Furthermore, the suitable physical transducer design must be the one that would allow measurements at high temperatures. In order to accomplish high temperature suitability, we will apply specially designed high temperature transducers. These devices are described on pages 14 and 15 of our publication U-777. Their brief description follows:

- 8.2.1 Direct contact high temperature transducers: These transducers are directly coupled to the hot test materials surfaces, and are suitable for continuous operation up to approximately 250°C (480°F). In general practice it is recommended that these transducers be used intermittantly because of the problems caused by couplant evaporation.
- 8.2.2 Delayed contact high temperature transducers: By utilizing field replaceable high temperature resistant delay materials on a modified direct contact high temperature device, these transducers can be operated in excess of 1000°C (1832°F). The purpose of delayed buffer is to provide protection to the active transducer. Coupling at such temperatures is generally a problem. This will be discussed in a separate section.

8.2.3 Dual T-R high temperature transducers: By combining the elements of high temperature designs in a and b above, it is proposed that suitable T-R types of transducers be designed to operate from room temperature to >1000°C (>1832°F).

It should be noted that before designing a suitable high temperature transducer for refractory corrosion measurement, its vital characteristics such as physical style, frequency, dimensions, etc., will be determined while experimenting at room temperature conditions, section #7. We have reasons to believe that the suitable style will be composed of a separate transmitter and receiver, i.e., the T-R style.

9. COUPLANTS FOR TRANSDUCER CONTACT

Those well-versed in the practice of high temperature ultrasonic applications, know not only the difficulties in obtaining a suitable and durable transducer design, but are also aware of severe problems of coupling such devices to hot surfaces. We propose the following to tackle the problems of high temperature transducer coupling.

9.1 From room temperature to 315°C

It is recommended that at room temperature measurements, vaseline or motor oil be used as couplant. For medium range temperature, i.e. up to approximately 315°C (600°F), high vacuum grease be used as coupling medium.

9.2 From 315°C to >1000°C

Reliable couplants for the examination of materials at very high temperatures are generally not public knowledge. Our proposal to circumvent this problem is to experiment with low melting glasses as couplants. Glass compositions rich in B₂O₃ content are considered good potential candidates for this purpose.

9.3 Alternative to coupling problems

For the successful achievement of the ultimate objectives of this project it is imperative that suitable high temperature transducers be designed. However, despite of the proposed methodology of high temperature coupling, which is expected to be suitable at laboratory scale, it may cause unwanted problems. We are obviously not sure about them at this stage.

A more affirmative solution to coupling problems is to "permanently bond water cooled delay lines to the glass contact refractory" at those locations where measurements are to be made. Ice-cold water can be circulated in such delay lines only prior to making ultrasonic measurements. This should be an important consideration after sufficient observations and data have been acquired under the objectives of this project.

10. TEST INSTRUMENTS AND HIGH TEMPERATURE FACILITY

As we stated earlier, ultrasonic measurements of refractories in general require utilization of relatively lower frequencies. Excitation of lower frequency transducers, from 50 to 500KHz, and amplification of their reflected or transmitted signals cannot be adequately accomplished by conventional ultrasonic instruments that are designed to operate into MHz frequencies. Lower frequency transducers require higher energies to drive them and more electrical gains for the amplification of their signals. Furthermore, it is also desired to excite such transducers with short-burst electrical impulses so that the amplified

transducer response corresponds to short pulses, section #6. For nearly three years Ultran has been in the process of developing such an ultrasonic system. It is known as "High Energy" HE-900 ultrasonic system, and its major objective is to facilitate measurements of highly attenuative media. We propose to use this system for experimental investigations of this project, while basing the final Refractory Corrosion Measurement System (RCMS) upon the modifications of HE 900.

10.1 HE-900, a high energy ultrasonic system

- a. RF Burst Pulser: RF burst is in the form of "packets" of -ve going "spike" pulses, which can be varied in energy from approximately -250 to nearly -900V. A mechanism for frequency tuning is also provided in order to optimize the transducer characteristics. Width of RF burst can be controlled by a separate switch depending upon the amount of energy required to propagate through a given medium at a given transducer frequency. The RF burst of HE-900 can be used from nearly 25KHz to 2.0MHz.
- b. Receiver/Amplifier: The receiver/amplifer of HE-900 features total gain of approximately 102dB and usable bandwidth from nearly 25KHz to 2.0MHz. Two types of RF displays are provided as per user preference, i.e., unrectified RF display and rectified full-wave. These are selectable from a separate switch.
- c. High Temperature Suitability: The current ambient temperature stability of HE-900 is from -10°C to 50°C. While we do not expect the need for further increment in its high temperature suitability for the laboratory experiments proposed here, for the RCMS it will applied in order to facilitate its use around the furnace areas.

10.2 Tektronix 2432 digital oscilloscope and PEP 301 system

Ultrasonic signals obtained from test refractory specimens by the applications of transducers with HE-900 will be displayed and analyzed by a Tektronix 2432 digital storage oscilloscope for time-of-flight measurements. Tektronix PEP 301 system controller will be used for data storage and computational purposes. A schematic layout for general purpose time domain ultrasonic analysis is shown in Fig. 19. This mechanism will be used throughout the experimental procedures described in the following sections of this proposal.

10.3 High temperature furnace

In order to conduct the high temperature evaluation of transducers as well as measurements on AZS refractory - with and without contact with molten glass - a bottom loading furnace with approximate cavity dimensions of $12 \times 12 \times 12$ in with a capability of operation up to 1600° C is proposed.

All equipment identified here is in the possession of Ultran, with the exception of a suitable furnace.

11. LABORATORY EXPERIMENTS SIMULATING FIELD CONDITIONS

As described in section #3, the principle of refractory thickness meter is is to measure and relate time-of-flight of ultrasound propagation through the test materials for thickness/corrosion correlations. However, for a reliable refractory corrosion measurement system three critical experiments must be performed:

- a. Mechanical integrity and acoustic stability of RCMS transducers at certain high ambient temperature,
- b. High temperature effects upon the velocity/thickness of AZS, and
- c. Measurement of AZS thickness, while it is in contact with molten glass.

The following sections describe the experimental procedures, triggered toward these evaluations.

11.1 Evaluation of RCMS transducers and measurement of temperature dependence upon the velocity/thickness of AZS:

An experimental set up for the determination of transducers high temperature suitability ("a" above) and systematic evaluation of AZS as a function of temperature and its ultrasonic velocity is shown in Fig. 19. Ultrasonic measurements as a function of temperature are proposed to be carried out in the increments of 50°C and up to 1500°C. By measuring the times-of-flight as functions of temperature, relationships shown in eq. 7, 8, and 9 will be established. Initially all high temperature thicknesses will be calculated by adding the appropriate thermal expansion factors to the room temperature values. This exercise will establish important relationships, such as thermal expansion as a function of velocity contraction, eq. 8, as well as the critical ultrasonic velocity as a function of temperature, eq. 7.

Assuming that the refractory temperature on the ambient side is 200C in the field environment, in the proposed experiments this value will be maintained. Suitable transducers will be directly or indirectly subjected to the hot refractory surface for 5 minutes. The test transducers will then be brought to room temperature. The thermal cycling of the transducers will be carried out between 10 to 15 times, during which periods, their mechanical integrity and acoustic behavior will be examined.

Dimensions and shapes of AZS samples for this experiment to be defined.

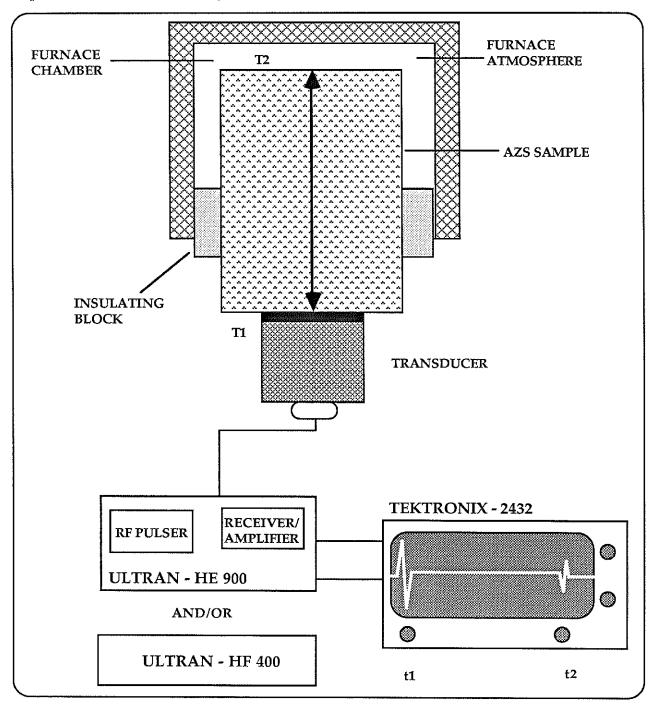
11.2 Measurement of AZS corrosion, while it is in contact with molten glass:

Results of experiments described in section #11.1 as per the sequence shown in section 3.2 will establish an ultrasonic method for AZS thickness measurement at high temperatures, such as the relation in eq. 9, 10, and 11. These relations being the foundation of corrosion measurements, times-of-flight through AZS, while its is in contact with molten glass, Fig. 20, will be monitored at a number of temperatures. These measurements will be directly converted into corrosion/thickness of glass contact refractory. It is further proposed that these experiments be performed on the glass contact AZS side representing a "degree of corrosion."

Dimensions and shapes of AZS samples for this experiment to be defined.

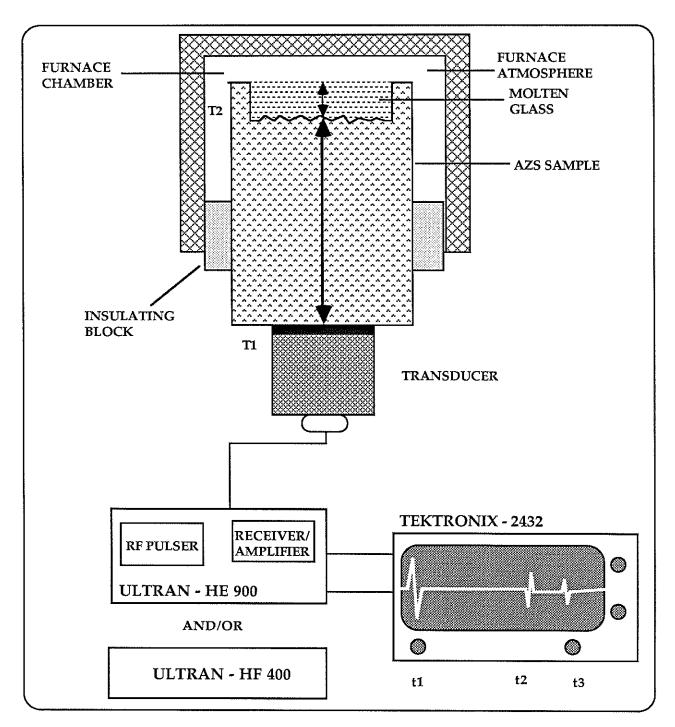
Mathematical analysis of data acquired from 11.1 and 11.2 will be performed in order to determine accuracy, resolution, and reliability of high temperature measurements. In all cases ultrasonically measured values of thickness/corrosion will be compared with the known values.

Fig. 19. A Schematci Layout for Transducer Evaluation and for the Measutement of Temeprature Dependence of Ultrasonic Velocity of AZS Refractory



- T1 Lower temperature side of the refractory 200 deg. C
- T2 Higher temperature side of the refractory
- t1 Interface of transducer and T1 refractory side
- t2 Interface of T2 refractory side and furnace ambient
- t2-t1 Time -Of-Flight (TOF) of ultrasound within the refractory

Fig. 20. A Schematci Layout for the Measurement of Molten Glass-AZS Interface.



- T1 Lower temperature side of the refractory 200 deg. C
- T2 Higher temperature side of the refractory
- t1 Interface of transducer and T1 refractory side
- t2 Interface of AZS and molten glass
- t3 Interface of molten glass and furnace ambient
- t2-t1 Time -Of-Flight (TOF) of ultrasound within the refractory
- t3-t2 TOF of ultrasound through molten glass.

12. DESIGN OF A PROTO-TYPE RCMS

The successful execution of work proposed in this project should lay the firm foundation for the design of a Refractory Corrosion Measurement System. Our RCMS design criteria are based upon the definition of suitable transducer characteristics, suitable transducer excitation and amplification system, frequency range of the amplifier, method of transducer application, calculation of refractory thickness from the time-of-flight, incorporation of temperature-dependent effects, and digital display of the corrosion. Identification and implementation of these specifications are the ultimate objectives of this proposal, thus achieving a functional measurement system..

The goal of RCMS will be to identify and isolate the time-of-flight corresponding to ultrasonic propagation through refractory thickness only. This will be dictated by the transducer application method as described in section #8.1. Should there be angulation of transmitted and receiving ultrasonic beams, due compensation will be provided to eliminate the effects of angulated beams. These functions will be achieved by electronic means. Should there be need for signal averaging or statistical computations for increased accuracy, they will be implemented accordingly in the electronic design of RCMS.

Section #3.2 dealt with the theory of temperature effects upon ultrasonic measurement of AZS, and section #11 described the experimental procedures for obtaining critical thermal expansion related effects upon the ultrasonic velocity of AZS. This data will quantitatively define temperature compensation factor to be included in the electronic brain of the RCMS.

At the time of writing of this proposal we believe that this system will be fully programmable and self-sufficient, containing a mechanism for temperature and velocity compensation factors. These concepts will facilitate the use of RCMS on a wide range of materials, whether they are at a temperature gradient, or not.

The proto-type RCMS is expected to weigh below 4.5kg and will also have a rechargeable battery operation provision. An idealized sketch of RCMS is shown in Fig. 21.

13. PERSONNEL

Ultran Laboratories is well equipped with respect to experienced and knowledgeable personnel. Together, we specialize in Materials Science, Engineering, & Technology, Ultrasonics Electronics, Systems Engineering, Transducer Technology, and Ultrasonic Applications Technology. The following individuals of Ultran Laboratories will play the key role in the execution of work identified in this proposal from start to finish. Since we are located in the vicinity of Penn State University we wish to take the advantage of the expertise of personnel of Ceramic Science and Materials Research Laboratory. Specifically, we wish to do so in the areas of high temperature experiments, transducer coupling, and high temperature effects upon critical piezoelectric materials. The Penn State personnel are to be used as Ultran's consultants. At the time of this writing we identify those Penn State individuals who we believe would be most valuable in producing the most affirmative results from the problems cited in this proposal.

13.1 Ultran Laboratories, Inc.

Mahesh C. Bhardwaj, Director R & D: Overall responsibility of the project, transducer design and room and high temperature experimental set up and design.

Charles J. Valenza, Director Transducer Technology, Transducer design, and manufacture of proto-types.

Arun Madhav, R & D Scientist, execution of room and high temperature ultrasonic measurements.

Anant S. Trivedi, Director Ultrasonic Electronics, design and manufacture of proto-type RCMS.

13.2 Pennsylvania State University

David J. Green, Professor Materials Science, high temperature experiments design and high temperature coupling compounds.

Amar S. Bhalla, Professor Solid State Science, high temperature effects upon piezoelectrics, and transducer coupling.

14. OTHER SECTIONS OF THE PROPOSAL

Information, relative to place/s of work, milestones, deliveries, and costs is available and will be discussed with those companies who would show strong interest in the subject matter described in this proposal.

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END TECHNICAL PROPOSAL

MCB:cbm