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Phase I Report

ULTRASONIC MEASUREMENT OF CORE MATERIAL TEMPERATURE

by

M. S. McDonough, L. C. Lynnworth and E. H. Carnevale

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

December 1967

CONTRACT NAS' 3-10284

Advanced Systems Division Dr. John C. Liwosz



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M. S. McDonough, L. C. Lynnworth and E. H. Carnevale

ABSTRACT

A pulse-echo ultrasonic system was used to determine temperature by measuring the round trip transit time in a Re wire sensor. Measurements on Re were conducted up to 6216° R in carbon-free vacuum, and up to 4800° R in a graphite environment. Above $\sim 3500^{\circ}$ R, enough carbon diffuses into the Re sensor in one hour to perturb the transit time/ temperature calibration. A sheath is therefore required, to avoid this calibration shift, and to permit operation beyond the Re/C eutectic. Temperatures have also been measured at two different locations on one line using two different modes of vibration.

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SUMMARY

The main object of the first phase of this work was to conduct research, design, develop, construct, and test an ultrasonic system capable of measuring temperature up to 5300° R for one hour, to an accuracy of 50° R, in a graphite/hydrogen/nuclear environment.

High temperature ultrasonic measurements were conducted as functions of temperature and frequency to determine the feasibility of using a rhenium sensor as the high temperature sensor. The velocity and attenuation were measured in carbon-free vacuum up to 6216° R. Measurements were also performed up to ~5000°R with rhenium in intimate contact with graphite, in order to determine its effect on ultrasonic propagation in the rhenium sensor. It was found that when carbon diffuses into rhenium, the sound velocity increases. This occurs at temperatures above 3500°R. Furthermore, the temperature dependence of sound velocity substantially decreases. To avoid this, and also to permit operation above the Re/C eutectic, a sheath is required. Sensor materials other than rhenium may also be required, for use to 5300°R.

Measurement of two temperatures greater than 3000[°]R at two different locations on the same line, using extensional and torsional waves, has also been accomplished by using the Joule-Wiedemann effect.

It is recommended that a sheath be used in Phase II to protect the sensor from the graphite/hydrogen environment. The sheath is necessary since temperatures up to 5300° R are to be measured.

INTRODUCTION

Statement of the Problem

One of the more important measurements required in nuclear rocket engine technology is the measurement of temperature. This measurement has proven to be extremely difficult because of the high temperatures involved ($\sim 5300^{\circ}$ R), because of compatibility problems with some of the materials involved (graphite and hydrogen) and because of the intense and sustained neutron and gamma fluxes. Additional difficulties stem from the high ambient noise, shock and vibration levels expected in some locations, the accuracy and response time required, possibility of temperature overshoot, high pressure, flow, accessibility and geometrical restrictions, etc.

Up to now, thermometry based on thermocouple developments has received the greatest attention, with respect to operating in the above nuclear rocket engine core environment. At this time, however, a number of thermocouple problems still remain unsolved.

Present Ultrasonic Approach

Ultrasonic measurements of temperature in a nuclear rocket engine may be applied in the core and also in the thrust chamber (Fig. 1). Our work on through-transmission gas thermometry is reported elsewhere (ref. 1-3), the most recent instrumentation development being the Pana-Therm Model 10K, which is designed to automatically measure transit time across the hot gas. Core thermometry, on the other hand, exploits the pulse-echo technique applied to a thin wire sensor. The rest of this report is devoted to the thin wire approach to ultrasonic thermometry in the core of a nuclear rocket engine.

Phase I Objective

The main objective of Phase I was to determine the maximum temperature that a rhenium sensor would be able to measure in a graphite environment. Initially, the goal for the temperature measurements was one hour at 5000° R. About two-thirds of the way through Phase I, however, the temperature objective was increased to 5300° R. Since this new temperature goal is above the rhenium-carbon eutectic, it follows that, if rhenium is to be used as the sensor, a sheath is required.

In the ultrasonic temperature measuring system, temperature is determined by measuring the round trip transmit time in a wire sensor. Transit time is determined by the pulse-echo method. That is, the ultrasonic signal is reflected from the beginning and end of the sensor. Usually, as temperature increases, transit time increases.

Besides changes in transit time, as temperature increases, the amplitude of the sensor's echoes usually decreases, due to attenuation. Attenuation becomes increasingly more severe as the temperature increases above 3000° R. Attenuation is characteristic of the material, vibrational mode, frequency, and dampening external to the wire. Usually, attenuation decreases as the frequency of the ultrasonic signal decreases; herein lies one basis for overcoming attenuation.⁵

We previously determined that rhenium appeared to be a suitable ultrasonic temperature sensor, since it does not form a carbide and its eutectic point is 4966° R. In the present contract, we investigated rhenium sensors with diameters ranging from 0.01" to 0.1" and with lengths varying from 1" to 5". The emphasis during this phase has been placed on 0.020" and 0.030" diameter sensors, since, for a reasonably small hole in graphite, the space required by a sheath and the necessary clearances indicates that the maximum diameter that a sensor can be is approximately 0.030".

In the present work, the ultrasonic line usually consists of a Remendur transducer wire, a tungsten lead-in wire, and the rhenium sensor (see Fig. 2). Joints were formed satisfactorily by flash butt welding. Echoes reflected by the various welds were essentially equal to that predicted by theory.

EXPERIMENTAL INVESTIGATIONS

Transducers

The coil used to launch and detect the ultrasonic wave is wound on the Remendur transducer wire. The ultrasonic wave is produced by the magnetostrictive effect. To increase the ultrasonic signal, a biasing magnet is positioned close to the transducer. The transducer coils used to obtain the following results were wound with #36 AWG copper wire. Sixteen feet of wire was used for every inch of coil length. Figure 3 is a plot of the pulse width of the signal vs coil length for extensional and torsional waves. The measurements were performed for each coil by varying the pulse width until the echo amplitude was maximized. It should be observed that the pulse width varies approximately linearly with the coil length. The curve begins to fall off for the $3^{\prime\prime}$ coil. This is probably due to flux leakage. Figure 4 shows the minimum sensor length (echoes from each end of the sensor do not overlap) vs coil length. The sensor length must increase linearly as the coil length increases. This effect can also be interpreted in terms of frequency by saying that the sensor length must increase as the frequency decreases. Satisfactory results were also obtained magnetostrictively, by winding a coil directly on the 0. 125'' diameter Remendur wire and connecting this to the same 0.09'' diameter sensor. Figure 5 shows a piezoelectric transducer which was used when the sensor has a diameter of ~0.09''.

Attenuation

Attenuation decreases as frequency, f, decreases, if the attenuation is proportional to fⁿ and n is greater than one. It was found that the attenuation for rhenium and tungsten was less at low frequency (coil lengths of 3" and 1-1/2") than for high frequency (1/4" coil). Figure 6 shows reflections from the ends of a two foot tungsten leadin at room temperature and at ~5400°R. The transducer coil had a length of 3". The signal from the end of the lead-in is readily identified. The attenuation of the ultrasonic signal increases rapidly above 3500° R. For a 1/4" coil it would have been difficult to identify the end echo. Figure 7 shows the ultrasonic signals produced by a 3" coil for a 5" rhenium sensor at room temperature and at ~5300°R.

The attenuation as a function of temperature for tungsten and rhenium is shown in Fig. 8. Attenuation of the ultrasonic signal was calculated by measuring the pulse heights at room temperature and at elevated temperatures, and then following the procedure described in NASA CR-72339, Figures 45c and 46.⁴ These measurements in W and Re infer that a line consisting of a two foot tungsten lead-in wire^{*} and a 5 inch rhenium sensor can all be heated to 5300° R, and the temperature of the sensor will still be measurable at a pulse width of ~10 µ sec (frequency of ~100 kHz).

^{*}The attenuation due to additional tungsten lead-in wire at temperatures less than 3500°R is negligible.

^{**}In this work the frequency is taken as the reciprocal of the pulse width.

Equipment

The equipment used in the various experiments includes the Pana-Therm Model 5000, the General Radio 1217-C Unit Pulser and the 1397-A Amplifier. A block diagram of the complete system is shown in Fig. 9. By using the pulse amplifier, the output impedance, polarity, and rise and fall times of the driving pulse can be varied. The echo amplitudes can sometimes be improved if the shape of the driving pulse is trapezoidal instead of square. This improvement was predicted theoretically by Rothbart and Rosenberg.⁷

The metal wires used in this work have been heated in vacuum by the self-heating technique. With this method, for example, rhenium was heated to its melting point, 6216° R. A photograph of the equipment used in a typical experiment is shown in Fig. 32 of reference 1. The thin wire is supported in a vertical position in an evacuated Vycor tube. As the wire is heated it tends to bow due to expansion. This expansion can become a problem if it results in the wire coming in contact with the tube. Bowing is avoided, however, by attaching a coiled piece of braided copper to the bottom of the sensor. The weight of the braid keeps the self-tested wire sufficiently straight.

Rhenium Sensors Self-heated in Vacuum

To determine the effects of graphite on rhenium at temperatures up to $\sim 5000^{\circ}$ R, it was first necessary to measure transit time in carbonfree rhenium as a function of the self-heating current and also as a function of temperature. (Similar measurements had been obtained in a preliminary manner in the previous contract.) Figure 10 is a plot of the transit time vs current for rhenium sensors of lengths 2" and 3" and diameters of 0.020". A 1" sensor did not have the same characteristics. This is attributed to the large temperature gradients present in such a short sensor. ^{*} Figure 11 shows a plot of current versus temperature for self-heating experiments in this laboratory and in other laboratories. The curves agree reasonably well. Figure 12 shows the resistivity of rhenium and other refractory metals as a function of temperature.

As explained previously, the temperature goal of Phase I was increased from 5000° R to 5300° R. Since this is above the Re-C eutectic

^{*} Gradients in self-heated W are shown in <u>Temperature Its Measurement</u> and <u>Control in Science and Industry</u>, Vol. 3, ed. Charles Herzfeld, pp. 523-534.

temperature a sheath is required to assure survival of rhenium. Therefore emphasis was placed on measuring propagation in rhenium in carbon-free vacuum, at a temperature of at least 5300° R. Table I lists the transit time, the clock time and the self-heating current corresponding to 19.5 amps. With the aid of Fig. 13 it was determined that the temperature was approximately 5300° R. Pyrometer readings were taken and the uncorrected temperature was found to be 4352° R. When this reading was corrected for the wall loss of the Vycor tube and the emissivity of rhenium, the corrected temperature was found to be $\sim 5380^{\circ}$ R. At this temperature, transit time remained constant for one hour. Transit time vs current measurements were performed several times and the results were the same. Having demonstrated the stability of rhenium at a temperature of at least 5300° R can be measured ultrasonically to the extent that a sheath prevents carbon diffusing into the sensor.

Rhenium Sensors in Graphite

Prior to increasing the temperature goal above the Re/C eutectic, indirect heating and self-heating were used to study rhenium in a graphite environment for temperatures up to 4966° R. For the indirect heating method, rhenium was placed inside a heated graphite sleeve. Indirect heating methods included (1) induction heating, (2) resistance heating of the graphite and (3) resistance heating of tungsten wire surrounding a graphite cylinder. Studies of graphite effects in rhenium were not completed with these indirect heating techniques because the self-heating technique was meanwhile found to be the easiest to execute in the time prior to the increase in the Phase I temperature objective. (Preliminary self-heating tests in graphite felt had also been conducted in the previous contract. ⁴)

In this phase, self-heating experiments were conducted initially using either powdered graphite or a solid cylinder of graphite intimately surrounding the rhenium sensor. When the sensor became white hot, that is, when its resistivity reached about $100 \ \mu\Omega$ -cm, the graphite began to shunt the current intended for heating the sensor. Due to shunting, the current became unstable and the sensors melted. To avoid shunting, graphite felt manufactured by Basic Carbon Company was used. This felt enabled us to perform measurements on rhenium in a graphite environment at temperatures up to the eutectic, $4966^{\circ}R$. Figure 14 shows the transit time as a function of current for different rhenium sensors that have been heated to $4800^{\circ}R$. From these figures it is seen that the transit time decreases as the length of time for which the rhenium is heated in graphite increases. On cooling, the average slope of the curve

6

has decreased by a factor of approximately two. The change in transit time and the resulting change in slope are attributed to the presence of C in the Re sensor. Tables II, III, IV and V give analyses of (1) a Re sensor before and after heating in graphite, (2) the amount of carbon that was found in sensors that had been heated in graphite felt, and (3) the graphite felt. These analyses show that a significant amount of carbon diffused into the sensor after it was heated in a graphite environment. According to the phase diagram of the rhenium-carbon system, the temperature at which 0.4 weight percent of C in Re is in equilibrium occurs at ~4450[°]R. Thus, the temperature of this sensor was at least 4450[°]R. After at least three hours, this sensor was reheated for at least one hour to the same temperature. The heating and cooling curves were found to be very similar. Figure 15 shows the transit time as a function of current for sensors that have been previously heated in a graphite environment for a period of at least one hour.

This similarity in the heating and cooling curves in Fig. 15 is attributed to the carbon that has diffused into the rhenium. Table IV shows that $\sim 0.35\%$ by weight carbon has diffused into the rhenium. When the sensor's surface was examined with a magnifying glass a film was found to have formed. The change in the transit time was plotted as a function of the heating time at a fixed current, and the results for the different sensors are shown in Fig. 16. Transit time does not change for approximately the first 10-15 minutes, but then it changes rapidly, finally approaching an asymptotic value. The rapid change in transit time for a fixed current is attributed to the rapid diffusion of carbon into the rhenium. As time increases, proportionately less carbon diffuses into the sensor, and this results in a decreasing change in transit time. After a period of approximately three hours the transit time approaches its asymptotic value within 2%. During the period of heating, the voltage across the sensor did not change and the conductivity of the sensors at room temperature before and after being heated in graphite remained essentially the same. This means that the current passing through the sensors did not change as the carbon diffused into the rhenium. It was found that a rhenium sensor could be heated to at least 3500[°]R in graphite without affecting the ultrasonic velocity. This result is important since it shows that a bare rhenium sensor can measure temperature in a graphite environment up to at least 3500°R for periods up to one hour. To date, the work on the feasibility of using an unprotected rhenium sensor beyond 3500°R in a graphite environment is inconclusive. Based on present information a sheath system will be used to protect the rhenium sensor from the graphite/hydrogen environment in the nuclear reactor, for operation to 5300° R up to one hour.

Joule-Wiedemann Effect

The measurement of two temperatures on the same line, using two modes of propagation, has been accomplished using the Joule-Wiedemann effect. When a longitudinal and a circumferential magnetic field are simultaneously applied to a long wire of a ferromagnetic material, the resultant lines of force form helices about the axis of the wire. If a magnetostrictive transducer is used to generate acoustic waves in the wire, it is found that extensional and torsional waves will be simultaneously produced. This is known as the Joule-Wiedemann effect. ⁸ In the following, we refer to the generation of torsional waves as the Wiedemann effect, to distinguish from the generation of extensional waves only, by the Joule effect.

An important condition necessary for obtaining the Wiedemann effect is that the magnitude of the circumferential field must be larger than that of the axial field at the surface of the wire. (The reverse condition holds somewhere inside the wire.)⁹ The equation for the circumferential field, H_{A} , can be written as

$$H_{\theta} = \frac{Ir}{2\pi a^2}$$
(1)

where I is the axial magnetizing current, r the distance from the center of the wire, and a the radius of the wire. The circumferential field will have its maximum value at the surface of the wire. This value must be larger than that of the pulsed axial field.

The method used to produce the Wiedemann effect is shown in Fig. 17. The wire is first demagnetized, and then magnetized in the circumferential direction by discharging capacitors through a longitudinal section of the wire. To maximize the circumferential field, the discharge circuit is connected as close as possible to the transducer coil. Measurements have been made in a line consisting of a magnetostrictive wire, a tungsten lead-in wire, and 2" and 7" tungsten sensors (Fig. 18). The temperature of each sensor was determined by measuring the velocity of the extensional and/or torsional wave in the appropriate sensor. Since extensional and torsional waves have quite different velocities, it is easy to differentiate between these two types of ultrasonic waves. Using the pulse echo technique, the two echoes associated with each sensor were observed on an oscilloscope. As the temperature of the sensor increased, the separation of the echoes increased (see Figs. 19 and 20). The sensors were self-heated in vacuum. The temperature of the first (2") sensor was determined by measuring the velocity of the torsional waves. Figure 19A shows torsional wave echoes when the 2" sensor is at room temperature ($\sim 530^{\circ}$ R) and also at 3000 °R. The second set of signals shows quite clearly that the velocity of the torsional waves has decreased at elevated temperatures. Figure 19B shows that extensional waves can also be used to determine the temperature of the same 2" W sensor.

Figure 20A shows torsional wave echoes in the 7" W sensor at room temperature and also at a temperature greater than 3500°R. The torsional echo from the end of the sensor is attenuated at these elevated temperatures. Figure 20B shows extensional wave echoes in the same 7" sensor, at room temperature and also at a temperature greater than 3500°R. The extensional end echo has not been severely attenuated. In all of the cases examined, the torsional waves were attenuated more than the extensional waves. Therefore, it is preferable to use the torsional waves to measure the temperature of the sensor closer to the transducer, while the temperature of the more distant sensor should be measured with extensional waves. Thus, by sequentially energizing torsional and extensional transducers, it has been shown that torsional waves can be used to measure a temperature of 3000[°]R in one sensor. while extensional waves can be used to measure a temperature of at least 3500[°]R in a second sensor, adjacent to the first, in the same wire. The preliminary data for attenuation in tungsten shows that the maximum distance the sensors can be separated is approximately one foot.

CONCLUSIONS AND RECOMMENDATIONS

Based on tests and analyses conducted in Phase I, it appears that an optimized system should consist of the following: Pana-Therm Model 5000, plus power amplification and pulse shaping circuitry, " a three inch transducer coil wrapped around a 0.06^{11} diameter Remendur wire, a 0.04^{11} diameter tungsten lead-in wire, and a five inch long, 0.03^{11} diameter W or Re sensor. Tests on W and Re have been conducted to at least 5400° R in carbon-free vacuum for one hour. Use of lower frequencies than in the previous contract overcomes attenuation even at these high temperatures. If a sheath can be developed to protect the sensor from the graphite environment it would appear that the ultrasonic system can measure temperatures up to 5300° R.

^{*} In this phase, the required power amplification and pulse shaping was provided by the General Radio 1217-C Pulses and 1397-A Pulse Amplifier. In the future, the required power and shaping will be incorporated into a new instrument, the Pana-Therm Model 5010. If required, these features can also be built into the Model 5000 prototype.

It is recommended that Phase II be initiated to determine the required sheath material and configuration.

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Clock Time-Minutes	Transit Time-µsec	Current-Amps
0	21.5	0
23	32.0	19.5
33	31.5	19.5
38	31.5	19.5
50	31.5	19.5
65	31.5	19.5
83	31.5	19.5
94	31.5	19.5

Rhenium sensor heated to $\sim 5300^{\circ}$ R

This table shows that over a period of one and one-half hours the ultrasonic velocity does not change at a temperature of $\sim 5300^{\circ}$ R.

|--|

Typical analysis^{*} of pure rhenium

Element	Parts per million
С	20
Si	3
н	5
Ν	10
0	10
Al	< 5
Co	< 3
Cr	< 3
Cu	< 2
Fe	≈70
Mn	< 2
Мо	< 25
Sn	< 2
Ti	< 2
Zi	< 2
All others	< 1

* Analysis performed by Cleveland Refractory Metals, Solon, Ohio.

	Analysis*	of rheni	um wire self-he	eated in	n graphite fe	elt	
Li	ND	Zn	ND	Sb	ND	Lu	
Be	ND	Ga	ND	Te	ND	Hf	ND
В	ND	Ge	ND	Cs	ND	Ta	ND
Na	ND	As	ND	Ba	ND	W	ND
Mg	VFT FT	Rb	ND	La		Re	H
A1	VVFT	Sr	ND	Ce		Os	ND
Si	VFT	Y		\Pr		Ir	ND
К	ND	Zr	ND	Nd		Pt	ND
Ca	VVFT	Nb	ND	Sm		Au	ND
Ti	ND	Мо	ND	Eu		Hg	ND
v	ND	Ru	ND	Gd		Tl	ND
Cr	VFT	Rh	ND	Tb		Pb	VVFT
Mn	VFT	Pd	ND	Dy		Bi	ND
Fe	FΤ	Ag	VVFT	No		${ m Th}$	
Co	ND	Cd	ND	Er		U	
Ni	VVFT	In	ND	Tm		P	ND
Cu	VFT	Sn	VVFT	Yb			
Key:							
ND — VVFT VFT FT	── Not Deteo	cted 001% 1%	T .01- L .1-19 M 1%- H >10%	1% % 10%			

Table III

* Analysis performed by Jarrell-Ash Co., Waltham, Mass.

T	a	Ъ1	е	IV
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Analysis of a rhenium sensor that was heated in graphite felt

		Weight $\%$ of C that diffused
	Sensor	into the sensor.
1)	20 mil Re sensor-Heated to 4600 ⁰ R for at least 8 hours	0.35
2)	30 mil Re sensor-Heated to 4600 ⁰ R for at least 3 hours	0.42

*Chemical analysis performed by Werby Laboratories, Inc., Boston, Mass.

	Analysis [*] envir	of the gra onment fo	phite feltu or the self-l	sed to pro- neated rhe	vide the gra nium sense	aphite ors	
	ND	Zn	ND	$\mathbf{S}\mathbf{b}$	ND	Lu	
	ND	Ga	ND	Te	ND	Hf	
	VVFT	Ge	ND	Cs	ND	Ta	
	VFT	As	ND	Ba	ND	W	
	FΤ	Rb	ND	La		Re	
	VFT	\mathbf{Sr}	ND	Ce		Os	
	FΤ	Y		\Pr		Ir	
	ND	Zr	ND	Nd		Pt	
	Т	Nb	ND	Sm		Au	
	FΤ	Мо	ND	Eu		Hg	
	ND	Ru	ND	Gd		Tl	
	FT	Rh	ND	Tb		Pb	
	VVFT	Pd	ND	Dy		Bi	
	FΤ	Ag	VFT	Но		Th	
	ND	Cd	ND	Er		U	
	VVFT	In	ND	Tm		Р	
	VVFT VFT	Sn	ND	Yb			
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T T	→ Not Det < 0001% . 0001%	ected	T. L. M. 1	01-1% 1-1% %-10%			

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*Analysis performed by Jarrell-Ash Co. Waltham, Mass.



- MEASUREMENT LOCATION

Figure l. Nuclear rocket engine.

0.060" diameter 0.040" diameter 0.020" diameter Remendur wire W lead-in wire Re sensor -**1''to** 5''---– 1' to 5'---* k ★ $\sim 1^{1}$ Transducer coil

Fig. 2. Ultrasonic line used to measure temperature. The ultrasonic echoes reflect from the beginning and end of the rhenium sensor. Dimensions shown approximate the typical values for the present program. See also Fig. 9 for instrumentation details.



Fig. 3. Pulse width of the ultrasonic signal as a function of the length of transducer coil.



Figure 4. Minimum sensor length that can be used as a function of the length of the magnetostrictive coil.





Mounting sub-assembly

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Figure 6. Echoes in a 2 foot by 0.03 inch diameter W wire at room temperature and at 5400°R, produced with a 3 inch transducer coil.







Room temperature

Figure 7. Self-heated rhenium wire, 0.020" dia by 5 inches in length. The coil used was 3 inches in length.





Fig. 9. Equipment used to transmit and receive ultrasonic signals









Figure 12. Electrical Resistivity of rhenium, tungsten, tantalum and molybdenum as a function of temperature.





AMPS



Fig. 14. Transit time as a function of the current used to heat the rhenium sensor in graphite felt. The change in transit time is attributed to the diffusion of carbon into the rhenium.





Fig. 15. Transit time as a function of self-heating current. The rhenium had been previously heated in graphite felt, and the small slope of the curve is attributed to the carbon that has diffused into the rhenium.



Fig. 16. Transit time of the ultrasonic signal vs the time that the sensor was heated in a graphite environment. The transit time approaches an asymptotic value, which implies that the carbon concentration has diffused to equilibrium.



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Figure 18. Ultrasonic line used to measure two temperatures along the same line.





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Figure 19. Ultrasonic echoes measure temperature in 2 inch W sensor



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Room temperature

> 3500[°]R





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Room temperature

$$> 3500^{\circ} R$$



Ultrasonic echoes measure temperature in the 7" W sensor. Figure 20.

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