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CORRELATION OF THE HEAT TRANSFER PROPERTIES AND ULTRASONIC TRANSMISSION PROPERTIES OF BONDS

by

Roberta Ann di Novi

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Roberta Ann di Novi

Metallurgy Division

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CORRELATION OF THE HEAT TRANSFER PROPERTIES AND ULTRASONIC TRANSMISSION PROPERTIES OF BONDS

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I. INTRODUCTION

The acceptance or rejection of a bonded element that has been tested by ultrasonic methods has been made on the basis of the ability of the bond to transmit or reflect ultrasonic energy with only occasional reference to destructive evaluation of the bond. The criteria for acceptance are often poorly defined, as the tests do not provide any information about the serviceability of the element, i.e., its ability to transmit heat, because it is not known what correlation, if any, exists between the heat transfer and ultrasonic transmission properties. No previous investigation has been made of this problem, although one group¹ has recently reported using "a form of frost testing in an attempt to correlate heat transfer with ultrasonic findings" as part of a nondestructive test on brazed joints. This program has sought to make ultrasonic bond tests more significant by establishing a relation between the heat transfer and sound transmission properties of bonds.

Two types of bonds, mechanical bonds in the form of copper braze-bonded specimens and roll bonds in the form of Borax fuel-plate sections, have been investigated.

II. THEORETICAL ASPECTS

To determine the thermal properties of bonded elements, the thermal-pulse method for measuring thermal diffusivity and thermal conductivity developed by Parker *et al.*² is being utilized. Its particular advantage is that measurements are made in such a short time that heat losses are negligible and consequently the boundary conditions are easily satisfied. In this method, the front surface of the specimen is uniformly irradiated by a very short pulse ($\geq 500 \mu\text{sec}$) of radiant energy supplied by a xenon flash tube. The temperature history of the back surface is continuously monitored by feeding the output of the thermocouple in contact with the back surface into a differential amplifying system and then into an oscilloscope. The oscilloscope trace can then be photographed.

For a perfectly insulated solid of uniform thickness L , which has instantaneously and uniformly absorbed a pulse of radiant energy, Q cal/cm², at its front surface, the temperature history at the back surface is described by

$$\frac{T(L, t)}{T_M} = 1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp\left(\frac{-n^2 \pi^2 \alpha t}{L^2}\right), \quad (1)$$

where

T_M = maximum back surface temperature, °C;

α = thermal diffusivity, cal/cm².

When $T(L, t)/T_M = 0.5$, that is, when the back-surface temperature has reached half its maximum value,

$$\alpha = 1.38 L^2 / \pi^2 t_{1/2}, \quad (2)$$

where $t_{1/2}$ is the time required for the back surface temperature to reach half its maximum rise. Thus, α is determined by a measurement of the sample thickness and the half rise time.

The thermal conductivity K of a material is

$$K = \alpha \rho C (\text{in cal cm/cm}^2 \text{sec}^\circ\text{C}), \quad (3)$$

where

ρ = density, g/cm³;

C = heat capacity, cal/g°C.

Since

$$\rho C = Q/LT_M, \quad (4)$$

the conductivity can be written as

$$K = \alpha Q/LT_M, \quad (5)$$

and the conductivity is determined when Q , the amount of energy falling on the front surface, is measured. Parker et al.² estimate the accuracy of the experiment as $\pm 10\%$.

This analysis is based on a solid plate, and specimens used in this experiment have one or two interfaces. To find even an approximate solution

for a layered media is exceedingly difficult, and the experimental results indicated that a good approximation was achieved by using the solid-plate solution. The temperature curve for the back surface showed, in most cases, no deviation from the theoretical curve. There was also no change in the measured values of the diffusivity and conductivity when the sample was turned over, reversing the front and back surfaces. Distortions to the curve occurred only when measurements were made over a region of greatly changing thermal properties, and such areas were not used in the final analysis. The thermal diffusivity and conductivity measured are composites of the diffusivities and conductivities in each layer, and by assigning a conductivity value to a particular bonded element, the implication is that this bonded specimen acts as if it had a conductivity of such value.

III. EXPERIMENTAL ASPECTS

A. Thermal Measurements

Meaningful measurements can be made only if all the required boundary conditions are met and the theoretical assumptions fulfilled. Measurements are made in such a short time (30 to 60 ms) that radiation losses are negligible at low temperatures. To reduce conductive heat losses, the sample holder was made of fired Lavite, a ceramic with a very low conductivity. The holder contacted the specimen only around its edges, and the specimen was held in place by small clips which obscured very little of the front surface. The condition of uniform illumination requires that only a small percentage of the total surface could be covered.

The condition of uniform illumination of the front surface was difficult to achieve with specimens as large as the ones used (19 mm square), and an optical system was introduced to focus the light from the flash tube onto the front surfaces. It was found that overexposed X-ray film placed at the front surface position gave a visual indication of the uniformity of the illumination. With an optical system, the specimen and thermocouple are a considerable distance from the flash tube and its associated circuitry, thereby eliminating the problem of large induced voltages that occur in the thermocouple circuit when the tube is flashed.

To decrease the thermal resistance, a spring-loaded 26 gauge Chromel-Alumel thermocouple, whose ends were sharpened to a fine point and separated by one mm, was placed in contact with the back surface. The thermocouple circuit was then completed through the back of the specimen, insuring that the temperature monitored was actually the back surface temperature. The thermocouple contact had to be maintained at a constant and maximum value, or errors occurred in both diffusivity and conductivity values. This was easily checked by switching an ohmmeter across the thermocouple to measure the circuit resistance. The spring-loaded thermocouple was mounted to a microscope-table positioner, enabling the thermocouple to be moved to any point on the back surface. Small scales on the

positioner provided for the determination of the coordinates of each point with respect to the sample boundaries.

The amount of heat falling on the front surface of the specimen is a constant when the distance between the flash tube and each sample remains the same and when the flash-tube input energy is constant for each discharge, that is, when the storage capacitor is charged up to the same voltage for each flash. The amount of heat absorbed by the front surface of each specimen may be kept constant by coating the front surface with a thin film of graphite. The maximum temperature rise of the back surface of samples which had been cleaned and recoated several times was found to be reproducible to 0.1°C .

The General Electric 524 xenon flash tube dissipated 2000 J in each flash. Maximum temperature rises of the back surface were about $4\text{--}8^{\circ}\text{C}$, and a Burr-Brown Operational Amplifier--Model 1303--amplified the thermocouple voltage by 1000 before it entered the oscilloscope differential preamp. A bucking voltage was introduced into the thermocouple circuit to provide a means for adjusting the position of the scope trace. Figure 1 is the block diagram of the apparatus, and Fig. 2 is the room-temperature setup.

The system was calibrated against copper and aluminum, which both have specific heats and densities that are well-known at room temperature. By use of Equation 4, Q as determined from the aluminum standard was

$$Q = 0.447 \text{ cal/cm}^2$$

Fig. 1

Block Diagram of Room-temperature Experimental Apparatus

Fig. 2. The Room-temperature Setup for Measuring Thermal Diffusivity and Thermal Conductivity

and from the copper standard was

$$Q = 0.451 \text{ cal/cm}^2.$$

A value of $Q = 0.449 \text{ cal/cm}^2$ was used. This calibration was periodically checked as the flash tube loses efficiency with use.

Figure 3 is a typical curve of the temperature rise of a back surface. To the left is the transient which occurs when the flash tube is triggered; it serves to provide a zero point for measuring time. A comparison of the shape of the curve with the theoretical curve will verify, for any experimental curve, whether or not the boundary conditions are satisfied. Figure 4 is the temperature history of a back surface taken at a sweep speed of 0.2 sec/cm . This verifies that the assumption of no heat losses during measurement is justified, as T_M stays constant for over 1.5 sec. A distorted curve results when there is a violation of boundary conditions, and such violations are readily detected. Figure 5 is the curve obtained for a condition of nonuniform illumination. There is "hot spot" at the center of the front surface of an Armco iron specimen.

Fig. 3

The Temperature Curve
for the Back Surface of
SS 304 at 20 ms/cm and
50 mV/cm

Fig. 4

The Temperature Curve
for the Back Surface of
SS 304 at 0.2 sec/cm
and 50 mV/cm

Fig. 5

A Distorted Temperature
Curve for the Back Surface
of Armco Iron due to Non-
uniform Illumination of the
Front Surface

The method requires the use of small specimens, and all specimens were 1.9 cm square. The six mechanical-bond specimens consisted of two 1.02-mm-thick copper plates which were braze bonded to form a 2.03-mm-thick element. The standard to which thermal and ultrasonic measurements could be compared was a 2.03-mm-thick solid copper plate. The five roll-bonded specimens were sections of Borax fuel plates which showed suspect areas when tested with the ultrasonic imaging system; standards were made from areas that indicated a good bond. The Borax fuel plate is 0.76 mm thick and has a 0.38-mm core of UO_2 dispersed in SS 304 and 0.19 mm SS 304 clad. Tests were made with both bonded interfaces of the fuel plate. Although it is possible to cut off the cladding and its bond to the core on one side of the specimen, this would reduce the thickness of the specimen to the point where one of the conditions of the flash method, i.e., the pulse duration must be short in comparison to the time it takes the heat to travel through the specimen, would probably not be met.

B. Ultrasonic Measurements

The setup used for ultrasonic measurements duplicated, as closely as possible, the typical setup for ultrasonic nondestructive bond testing plates. A through-transmission method was utilized and tests were made at 0.5, 1.0, 2.25, 5.0, and 10.0 Mc. Branson Type ZK transducers were used in conjunction with a Sperry Type UR-480 Reflectoscope and Sperry Type RA-453 Monitor. The output of the monitor was fed into one input of a dual-trace oscilloscope preamp and the output of the monitor marker stage into the other input; the scope was triggered from the monitor. The amplitude of the transmitted pulse could then be accurately measured on the oscilloscope, and additional amplification of the pulse could be obtained from the oscilloscope amplifiers rather than the Sperry amplifiers, whose characteristics were not regarded as having sufficient linearity for these measurements.

The specimens were only 1.9 cm square, and the edge region causing sound scattering was a fairly large proportion of the sample area. As it was found that the scattering was a minimum from the specimen-water boundary, a Lucite holder was designed which secured the specimen only along 2 mm of its lower edge. The system was calibrated so that the position of the area of the specimen through which the ultrasonic beam passed was known.

Results for the solid, i.e., perfect bond, copper specimen indicated that the scattered sound from the edges of the specimen did not interfere with the transmitted pulse only in the center (7.0 mm^2) area, and only measurements in this region are meaningful. Consequently, ultrasonic measurements were made under two conditions. Both the sender and receiver transducers were collimated for a 1.0-mm-diameter beam, and each specimen was scanned across its complete surface in "points" 1.0 mm apart.

The amplitude of the transmitted pulse at each point was measured. Then, the collimation was changed to limit the beam to a 6.0-mm diameter, and the amplitude of the transmitted pulse in the center (6.0 mm²) area of the specimen was measured.

IV. EXPERIMENTAL RESULTS

A. Copper Braze-bonded Specimens

Radiographs of the copper specimens were made before any tests were made. These radiographs indicated regions where the bonding was changing greatly and regions of complete nonbonding due to the absence of any solder. They also aided in the selection of the specimens for tests, and, consequently, measurements were made on specimens with very different thermal properties. The thermal diffusivity of the copper standard is

$$\alpha_{\text{std}} = 1.14 \text{ cm}^2/\text{sec},$$

and the conductivity is

$$K_{\text{std}} = 0.922 \text{ cal cm/cm}^2\text{sec}^\circ\text{C}.$$

Values of the bonded specimens varied from

$$\alpha = 1.00 \text{ cm}^2/\text{sec};$$

$$K = 0.873 \text{ cal cm/cm}^2\text{sec}^\circ\text{C}$$

for the best bonded specimen that could be made to

$$\alpha = 0.0090 \text{ cm}^2/\text{sec};$$

$$K = 0.0083 \text{ cal cm/cm}^2\text{sec}^\circ\text{C}$$

for a specimen (No. 23) whose two sections were separated by an air gap and which were held together only by the shearing forces along the edges where the specimen had been cut from a larger section.

Figure 6a is a plot of the diffusivity and conductivity values for one of the better bonded specimens (No. 11-2A). The radiograph of the specimen (see Fig. 6b) illustrates how the variation in the radiographic density agrees with the variation in thermal properties. Figure 7a is the plot of diffusivity and conductivity for a specimen (No. 23) whose accompanying radiograph (Fig. 7b) shows it to be braze bonded only in one small section--the upper right-hand corner.

43136

Fig. 6a. Plot of Thermal Diffusivity and Thermal Conductivity for Copper Braze-bonded Specimen No. 11-2A

Fig. 6b. Radiograph of
Specimen No. 11-2A

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Fig. 7a. Plot of Thermal Diffusivity and Thermal Conductivity
for Copper Braze-bonded Specimen No. 23

Fig. 7b. Radiograph of Specimen No. 23

For each sample studied, the average value of the diffusivity ($\bar{\alpha}$) and of the conductivity ($\bar{K}$) in the central region was calculated, and these values were related to the standard by dividing the average value $\bar{\alpha}$ or $\bar{K}$ by the value of α or K for the standard. These values are given in Table I.

TABLE I. Values of the Thermal Diffusivity and Thermal Conductivity for Copper Braze-bonded Specimens

Specimen No.	$\bar{\alpha}(\text{cm}^2/\text{sec})$	$N_{\alpha}^{(a)}$	$\bar{K}\left(\frac{\text{cal cm}}{\text{cm}^2\text{sec}^\circ\text{C}}\right)$	$N_K^{(b)}$
11-2A	0.845	0.741	0.705	0.765
34	0.787	0.690	0.678	0.735
25	0.326	0.286	0.282	0.305
16	0.295	0.259	0.261	0.283
21	0.078	0.068	0.0661	0.0717
22	0.0443	0.0389	0.0398	0.043
23	0.0309	0.0271	0.0264	0.0286

$$(a)N_{\alpha} = \bar{\alpha}/\alpha_{\text{std.}}$$

$$(b)N_K = \bar{K}/K_{\text{std.}}$$

Because of scattering and attenuation effects, meaningful ultrasonic measurements could be made only at frequencies of 1.0, 2.25, and 5.0 Mc. Table II gives the amplitude of the transmitted pulse for the 7 specimens and for the standard in the central 7.0-mm² region.

TABLE II. Values of the Amplitude of the Transmitted Ultrasonic Pulse at 1.0, 2.25, and 5.0 Mc for Copper Braze-bonded Specimens in 1-mm² Areas

Specimen No.	A (Volts) at 1.0 Mc	$N_A^{(a)}$	A (Volts) at 2.25 Mc	N_A	A (Volts) at 5.0 Mc	N_A
Standard	2.0	1.0	1.0	1.0	1.0	1.0
11-2A	1.70	0.85	0.125	0.125	0.0465	0.0465
34	1.64	0.82	0.12	0.12	0.0450	0.0450
25	0.850	0.425	0.075	0.075	0.035	0.035
16	0.50	0.25	0.03	0.03	0.02	0.02
21	*		*		*	
22	*		*		*	
23	*		*		*	

*No ultrasonic transmission.

$$(a)N_A = A/A_{\text{std}} - \frac{\text{amplitude of transmitted pulse for specimen}}{\text{amplitude of transmitted pulse for standard}}$$

These results are graphically illustrated in Fig. 8, in which N_A is plotted versus N_K for the three frequencies. As α is proportional to K , N_A versus N_{α} would have the same relationship.

43137

Fig. 8. Normalized Ultrasonic Transmission vs Normalized Average Thermal Conductivity for Copper Braze Bonds

As the "point-by-point" ultrasonic measurements required the masking of the ultrasonic beam to a 1-mm diameter, the incident energy was greatly decreased and a "no transmission" level was reached at points whose thermal diffusivity and conductivity was much higher than the cutoff values when measurements were made over the 7-mm² area. Also, accurate measurements could not be made at 5.0 Mc. Figures 9 and 10 are plots of N_A versus N_K for 1.0 and 2.25 Mc, and represent the combined data from specimens Nos. 11-2A, 34, 16, and 25.

These graphs do not include those points where the temperature curve of the back surface was distorted. Such regions were readily located on the radiographs, and were areas of rapidly changing intensity or areas that were only partially bonded with solder, and hence, were areas of greatly varying bond quality. It had been previously established³ that radiographs give a good qualitative measurement of bond quality for brazed copper

Fig. 9
Normalized Ultrasonic Transmission at 1.0 Mc in 1-mm² Areas vs Normalized Thermal Conductivity in 1-mm² Areas for Copper Braze Bonds

43138

43135

Fig. 10. Normalized Ultrasonic Transmission at 2.25 Mc in 1-mm² Areas vs Normalized Thermal Conductivity in 1-mm² Areas for Copper Braze Bonds

plates. Ultrasonic measurements made in these regions also resulted in a somewhat distorted pulse. Had such points been included in Figs. 9 and 10, they would have resulted in scattering of the points, primarily because of the wide variations in the amplitude of the transmitted pulse through such regions.

It is evident that the amplitude of the transmitted ultrasonic pulse decreases with a decrease in heat transfer properties, i.e., thermal diffusivity and thermal conductivity, and that this relationship is strongly frequency-dependent. The rate of loss of ultrasonic transmission increases as the wavelength decreases.

Destructive evaluation of several of the copper specimens revealed that variations in the bond quality occurred for two reasons. There was in some regions a complete absence of solder. This is particularly evident in the radiograph of specimen 23, which contains solder only in one small spot. Specimens 21 and 22 are similar to 23 in that they contain almost no solder. The thermal resistance of the two contacting surfaces was extremely high, and the resulting conductivity values were extremely low. Variations of thermal conductivity, or more precisely, here, the contact conductance of the interface, are due to changes in the thermal resistance which in turn depends on:⁴

1. flatness of the contact areas;
2. nature of materials in contact;
3. contact pressure;
4. roughness of contact surface;
5. material in voids (thermal conductivity coefficient; pressure in case of gases);
6. state of oxidation of contact surfaces.

Through such specimens there was no ultrasonic transmission--all of the ultrasound was reflected at the interface at all test frequencies.

Other specimens, e.g., No. 11-2A, showed light spots that appear as "porosity" on the radiograph. These spots were much darker than the nonbond regions of No. 23 and also exhibited better thermal properties. These regions contained solder, but were not well-bonded in most cases because the solder did not properly wet the surfaces. These small areas of nonbonds, appearing as defects to the sound wave, would naturally have a frequency-dependent ultrasonic transmission with the transmission decreasing as the wavelength increased.

It had been previously established³ that a correlation can be established between the amplitude of the reflected echo from the back surface (which is proportional to the transmitted pulse) and the tensile strength of braze-bonded copper plates. Consequently, these studies have justified the nondestructive ultrasonic method of testing brazed bonds when the concern is directed to either the strength or thermal properties of the bond.

B. Roll-bonded Specimens

The Borax fuel plates from which specimens were made for this study had been previously tested and passed by the through-transmission method at a frequency of 2.25 Mc. The plates, however, were then later tested by the ultrasonic imaging system⁵ utilizing Lamb waves, and suspect areas were indicated. Five specimens were made from the suspect areas, and several specimens were made from other areas to serve as standards. Both thermal and ultrasonic measurements were made in the same manner as for the brazed specimens.

Thermal measurements on three Borax standards at three different points each gave values

$$\alpha_{\text{std}} = 0.0285 \text{ cm}^2/\text{sec};$$

$$K_{\text{std}} = 0.0315 \text{ cal cm/cm}^2\text{sec}^\circ\text{C}.$$

Table III gives the values for the average diffusivity and conductivity in the central 5.0-mm² area of each specimen. Although five out of six of the values are within 10% of the standard, which is the limit of accuracy of the experiment, it is, nevertheless, believed that there are definite but small differences in the thermal properties of the specimens, as the reproducibility of the measurements was found to be 2%.

TABLE III. Values of the Thermal Diffusivity and Thermal Conductivity for Borax Roll-bonded Specimens

Specimen No.	$\bar{\alpha}(\text{cm}^2/\text{sec})$	$N_{\alpha}^{(a)}$	$\bar{K}\left(\frac{\text{cal cm}}{\text{cm}^2\text{sec}^\circ\text{C}}\right)$	$N_K^{(b)}$
Standard	0.0285	1.0	0.0315	1.0
10A	0.0260	0.910	0.0286	0.907
8B	0.0279	0.982	0.0302	0.958
12	0.0261	0.919	0.0290	0.920
9A	0.0251	0.880	0.0277	0.879

$$(a)N_{\alpha} = \bar{\alpha}/\alpha_{\text{std}}.$$

$$(b)N_K = \bar{K}/K_{\text{std}}.$$

Sound scattering was more severe than for the copper specimens, and ultrasonic measurements had to be confined to the center 5.0 mm². Ultrasonic measurements in the central region were made by masking the sound beam to an effective 5-mm² area. The amplitude of the transmitted pulse through this central region was the same value (with a 1% variation in amplitude) for all the standards and all the specimens at frequencies of 1.0 and 2.25 Mc.

"Point-by-point" measurements were then made by masking the beam to a 1-mm diameter and scanning the entire back surface of the specimens. Measurements were made only at 2.25 Mc. Figure 11 is a plot of N_A versus N_K where

$$N_A = \frac{A}{A_{std}} = \frac{\text{Amplitude of transmitted pulse for specimen}}{\text{Amplitude of transmitted pulse for standard}}$$

and includes points from all five specimens.

43201

Fig. 11. Normalized Ultrasonic Transmission at 2.25 Mc in 1-mm² Areas vs Normalized Thermal Conductivity in 1-mm² Areas for Borax Roll-bonded Fuel-plate Specimens

By masking the beam, it was possible to achieve better resolution and, consequently, in some cases a decrease of ultrasonic transmission occurred with a decrease of thermal conductivity. These changes were in most cases so small that it would be unlikely if the ultrasonic test were made at a normal scanning speed that they would be detected at all. This would agree with the previous results made by the through-transmission method at normal scanning speeds and with a masked beam of about 1.5-mm diameter.

There are also a number of points where a decrease in thermal conductivity is not accompanied by any change in the ultrasonic transmission. It is possible that the thermal conductivity values do not represent real differences unless the value is greater than 10%, and it is also possible that the cause of the change in thermal properties could not be detected by the through-transmission method.

No destructive evaluation was made of these specimens, but destructive evaluation was made earlier of several Borax specimens which gave ultrasonic indications similar to the ones obtained on the Borax specimens used in this investigation when they were tested with the ultrasonic imaging system. In only one case was a true nonbond found; other suspect regions contained only large particles of UO_2 or a larger concentration of UO_2 particles.

It is most probable that the conductivity losses were due to variations of particle size or particle concentration of the dispersed UO_2 . This would always be detected by the thermal method but not always by the ultrasonic through-transmission method. A large UO_2 particle whose major axis is parallel to the sound direction would cause little, if any, change in the amplitude of the transmitted sound, but yet would substantially affect the thermal conductivity. The ultrasonic imaging method is a "point-by-point" scan, and the sensitivity of Lamb waves is greater. These two factors would account for the fact that suspect regions did not always give indications when tested with the through-transmission method.

This thermal method of testing bonds proved itself to be very sensitive to small changes in thermal properties. The requirements of this method, e.g., small sample size, satisfying of boundary conditions, and the relatively slow scanning speed, would exclude this method from practical consideration. It has, however, illustrated the sensitivity that can be achieved by thermal methods when a fast detector is used--temperature equilibrium was reached on the back surface of the copper braze bonds in 20 ms to, in the worse case, 0.1 sec. In our case, the area over which the measurements were made could be made as small as desired simply by putting the two pointed ends of the thermocouple closer together; thus, smaller and smaller defect areas could be found. The choice was made at 1.0 mm^2 because this was the smallest ultrasonic beam size feasible for testing.

Unless a definite relationship is established between the thermal conductivity and the ultrasonic transmission for each particular bonded element, it is not, of course, possible to predict the exact heat transfer properties from the ultrasonic properties. The situation can be generalized by stating that a decrease in the amplitude of the transmitted pulse always signified a loss in thermal conductivity both for the braze and roll bonds, but that, depending on the resolution and sensitivity of the ultrasonic system, there will be small areas of poorer thermal properties that cannot be detected by ultrasonic through-transmission method.

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