METAL DIFFUSION BONDED TRANSDUCERS FOR RESONANT ULTRASOUND

SPECTROSCOPY (RUS)

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Ultrasonic transducers for Resonant Ultrasound Spectroscopy (RUS) need to be produced in a variety of sizes and designs in order to optimize the signals, minimize the effect on the object being measured and deal with odd shaped objects, limited access, and different environments.[1] This continuous requirement to build new transducers for different projects led to the construction technique shown in fig. 1 . A piezoelectric element, selected according to the frequency range which is to be covered and the size of the sample, is glued to both an inertial backload and a support, usually a metallized kapton (plastic) diaphragm. Electrical connections to the piezoelectric element are also made with thin metallized kapton with a signal connection glued between the piezo element and the backload and a ground connection made via the diaphragm at the front.



Figure 1. Construction of a LiNbO3 / diamond transducer using epoxy.

Advantages of this design are 1) ease and rapidity of fabrication, it takes less time to assemble than for the epoxy to set, 2) the diaphragm acoustically decouples the transducer from the shell and acts as a shielding ground, 3) with good choice of transducer and backload a unit can be made which has little or no internal resonances in the frequency range of interest (our LiNbO₃ and diamond units have a lowest mode of about 4 MHz). Disadvantages are 1) often the electrical connections are capacitive because of the insulating properties of the epoxy layers - not a problem at MHz frequencies, but causes weak responses at lower frequencies, 2) "soft" epoxy and plastic layers between piezo and backload degrade performance by permitting unwanted modes of vibration in the transducer unit, 3) the epoxy cannot withstand high temperatures and small area bonds (such as the small ring of connection to the diaphragm, fig. 1) are weak. Strength can be improved, as in many commercial transducers by "potting" the whole assembly in some damping material, such as epoxy which also reduces the effect of any internal resonances. Unfortunately this also has the effect of reducing the response of the transducer and increasing the inertial effect of the transducer on the object being measured. Disadvantage 2) is of particular concern for doing NDT and basic materials science because peaks from modes of the transducer are difficult for computer programs (not to mention experienced operators) to distinguish from peaks produced by the object under test. If the piezoelectric disc is attached rigidly to a stiff, flat backload, its flexural modes are raised to frequencies near those of the bare backload, but a layer of relatively soft epoxy and plastic will not provide strong enough coupling to shift these modes to high frequencies, so they may appear as unwanted peaks in the spectrum of an object. The major problem is the epoxy it is non-conductive, plastic, degrades with time, and cannot handle many environments (such as ultra high vacuum and high temperatures), although epoxy-glued transducers seem to perform well at low temperatures.

A solution has been developed at the Los Alamos National Laboratory (LANL).[2] In essence it replaces all of the epoxy joints with metal bonds. When two clean metal surfaces (i.e. free of oxides and other contaminants) are brought into contact under pressure and the temperature is raised above some threshold (dependent on the metal) metal atoms will diffuse across the contact boundary removing the surface contact interface and effectively bonding the two metal parts into one part - the surfaces vanish into the interior of a single piece of metal. The components of a transducer unit can be coated with metal, either electrochemically or by vacuum evaporation and the resulting metal surfaces may be diffusion bonded together, forming a conductive, strong, and durable joint, thinner than any glue-and-plastic joint. This thin and rigid bond is capable of adequately coupling the rigid backload (such as diamond!) to raise the lowest mode to one consistent with the theoretical composite object, making such transducers as free as possible of unwanted low modes of the active element. This bonding technique is used in a few special industrial applications (e.g. titanium turbine blades) - the conditions of surface cleanliness, and the pressures and temperatures mean it is difficult to apply, and indeed just the temperatures needed to mobilize atoms in most metals would exceed the Curie temperature of the common piezoelectric ceramic materials (PZT-5, $T_c \sim 350^{\circ}$ C) destroying piezoelectricity. The "noble" metals -copper, gold and silver, however with melting points near 1000° C can be bonded with acceptable values for pressure and

temperature, provided the surface cleanliness condition can be met.[3] Copper oxidizes readily and special conditions (vacuum, inert atmosphere) must be applied to maintain clean metal surfaces. Gold is inert and is suitable for bonds, but the temperature needed is at the high end of our allowed scale, around 300° C. Silver has the unusual property that at 150°C the metal will dissolve its own oxide layer into the bulk, providing a clean surface. Bonding can thus be carried out in air near 200° C, making silver our primary choice for a bonding material, although we have also developed the capability for bonding other metals.

Initial experiments with silver diffusion bonding attached PZT-5 ceramic piezoelectrics to alumina backloads in air with electrochemically deposited films. A sequence of bonds in various sizes of transducer were made to determine the optimal bonding pressure and time [4,5] For a "safe" temperature of 200°C or slightly above, and a bonding time of 20 minutes, the bonding pressure to produce good bonds varied from 15,000 psi for the largest transducers (1/2" diameter) to 22,500 psi for the smallest (1/16" diameter). Several transducers were successfully fabricated and showed excellent response.[4] All bonds could be made simultaneously and the transducer casing and support were easily made mechanically robust. All of these transducers were made with piezoelectric ceramics of 1mm or greater thickness, which means the unit's internal compressional resonances may start to interfere with measurements at as low as several tens of kHz for the bigger ones and a few hundreds of kHz for the smaller ones. These transducers certainly cover the range useful for most NDT applications where parts are usually relatively large and thus have low resonance frequencies. A comparison of the low modes of a "good" glued transducer and a metal bonded transducer is shown in fig. 2. There are no components in these units which are subject to deterioration with time, they will function in environments where solvents are present, and direct DC electrical connections are made, reducing low frequency noise pickup.



Figure 2. Mechanical response of epoxy bonded and metal bonded transducers of similar size.

For basic materials science and for NDT of smaller components we also improved on the performance of our original glued Lithium Niobate / Diamond transducers which use 0.004" thick ,1mm to 1.5mm diameter LiNbO₃ discs. The Curie temperature of LiNbO₃ is $T_c = ~1100^{\circ}$ C although for reasons discussed later it is only an effective transducer up to about 500°C. Nonetheless these temperatures would be a considerable advance on the present capabilities.

To work on these smaller transducers we have constructed a UHV system to vacuum-evaporate metal films, press them into contact under vacuum and heat the surfaces to the required temperature. The vacuum system enables us to use clean, unoxidized surfaces of any metal which can be evaporated, but only one bond at a time can be made. The evaporation source coats upward facing surfaces of the piezoelectric element and backload and the two freshly coated surfaces are placed in contact. A linear motion feedthrough then applies the required force while a heating element heats the interface. The vacuum is held at 10⁻⁶ Torr or better during this process so at worst only a few atomic layers of oxide could form. Ideally the vacuum is good enough for less than a monolayer to form. The thin LiNbO3 discs are quite fragile so we use considerably less pressure than used with the PZT ceramic discs, but because the Curie point is higher we can use a higher temperature to increase the diffusion length. Using silver films of 2,000Å to 5,000Å thick, bonding at 250°C for 20 minutes and applying a pressure of ~2,000 psi we can reproducibly make a transducer unit comparable in size to our glued units, but with superior properties. Measurements made on the electrical response of metal diffusion bonded transducers and ,for comparison, bare piezo discs and a commercial damped transducer, verify the idea of removing unwanted low frequency modes from the bonded unit.



Figure 3. Electrical responses of a) a bare $LiNbO_3$ disc, b) a similar disc metal diffusion bonded to an alumina backload.



Figure 4. Electrical response of a) a commercial transducer, b) a metal bonded PZT/Alumina transducer.

Fig. 3a shows the electrical response of a bare 30 MHz compressional mode LiNbO₃ disc several modes are visible well below the fundamental compressional mode. Fig. 3b shows the response of a similar disc silver-diffusion-bonded to a backload made of alumina. The lowest modes of the piezo disc have been suppressed - while bonding to one side of a backload shifts the fundamental mode down to where it is just becoming visible at 25 MHz. Fig. 4 shows similar scans for a) a commercially available transducer and b) a diffusion bonded PZT/Alumina transducer made from similar components, in the range 0.3 MHz to 6 MHz. The metal bonded transducer shows a marked advantage. To mount the small transducer unit so that connections can be made for RUS measurements, we obtained some 0.001" thick stainless steel mesh, produced by photolithographically masking and etching a flat sheet to have 200 square holes per inch. This mesh replaces the kapton diaphragm. We bonded the piezo side of the unit to the mesh, but the area and shape of the bond allowed the transducer to be "peeled" off the mesh too easily. We circumvented this problem by mounting the transducer between two meshes and making a diffusion bond on both sides, as in fig. 5. Even before making these bonds the transducer provided excellent responses, relying just on the pressure contacts to the meshes. This design can be made entirely of high temperature materials and is much more durable than the glued kapton/epoxy units - partly due to the continuous mesh diaphragm, and makes good DC contacts for both grounding and signals. There is also another advantage to the mesh which is that the slightly rough surface helps to hold samples in place without removing the essentially point contact nature[1] of the mount.



Figure 5. Construction of a high temperature metal bonded transducer.



Figure 6. Behavior of a resonance peak in Ni near the Curie temperature.

Two of these transducers have been used in a high temperature RUS setup to observe the behavior of the modes in pure nickel. Nickel has a ferromagnetic Curie temperature of 358° C. The transition is clearly visible in fig. 6. These transducers are limited to about 500° C even though the LiNbO₃ has a Curie temperature of 1150° C because the elevated temperature excites free charges in the material reducing the resistance of the device[6], which effectively shorts out the charges induced by the piezoelectric response.

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