

# HTS THIN FILMS: PASSIVE MICROWAVE COMPONENTS AND SYSTEMS INTEGRATION ISSUES

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## ABSTRACT

The excellent microwave properties of the High-Temperature-Superconductors (HTS) have been amply demonstrated in the laboratory by techniques such as resonant cavity, power transmission and microstrip resonator measurements. The low loss and high Q passive structures made possible with HTS, present attractive options for applications in commercial, military and space-based systems. However, to readily insert HTS into these systems improvement is needed in such areas as repeatability in the deposition and processing of the HTS films, metal-contact formation, wire bonding, and overall film endurance to fabrication and assembly procedures. In this paper we present data compiled in our lab which illustrate many of the problems associated with these issues. Much of this data were obtained in the production of a space qualified hybrid receiver-downconverter module for the Naval Research Laboratory's High Temperature Superconductivity Space Experiment II (HTSSE-II). Examples of variations observed in starting films and finished circuits will be presented. It is shown that under identical processing the properties of the HTS films can degrade to varying extents. Finally, we present data on ohmic contacts and factors affecting their adhesion to HTS films, strength of wire bonds made to such contacts, and aging effects.

## I. INTRODUCTION

The desirable properties and potential benefits of the high-temperature-superconductors (HTS) have been well established [1]. Numerous published papers on both the basic properties of the materials and their application in prototype circuits have demonstrated that significant improvement can be made in certain components and systems by including HTS [2-4]. As efforts continue towards the inclusion of HTS-based circuits in working systems, attention is turning to issues regarding the quality and reliability of the HTS components. These include uniformity and repeatability of the starting HTS films and substrates, stability of the material through processing and assembly, formation of ohmic contacts to the HTS films and the ability to bond reliably to these contacts. Gauging the quality of a given HTS component or process is a subjective matter dependent upon the intended application and operating environment. While components are routinely produced for laboratory demonstrations and experiments, the more stringent demands of components for real systems require a higher level of material and processing control.

The Naval Research Laboratory's High Temperature Superconductivity Space Experiment II (HTSSE-II) has recently provided an opportunity to assess the ability of current HTS technology

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to produce components for a space qualified sub-system. For HTSSE-II a hybrid semiconductor-superconductor microwave receiver-downconverter was designed and assembled with a level of space qualification conforming to a class 'D' space experiment [5]. This qualification process followed MIL-STD-883 and covered basic issues such as shock and vibration, bond pull strength, die shear strength and hermiticity. Basic concerns such as these are of interest in any systems application.

The topics covered in this paper relate primarily to the quality and durability of the HTS films and devices as they are subjected to the various fabrication and assembly steps necessary to produce a packaged device. We present data on the uniformity of the starting material and the resultant spread in the material's properties as it is subjected to the various processing and assembly steps. Also, we examine the ohmic contacts to the HTS material in terms of the adhesion to the HTS material, the strength of wire bonds made to those contacts and the contact resistivity ( $\rho_c$ ). The data presented here are not the result of a systematic study but rather are observations recorded in the course of producing the HTSSE-II module in our laboratory. Hence, aspects of this work may not be fully complete. It is hoped, though, that the material covered here will provide a guide to the further development of HTS devices for practical systems.

## II. HTS FILM UNIFORMITY/REPEATABILITY

As HTS moves towards insertion into practical systems, uniformity of the components is of major importance; HTS devices must have repeatable and predictable properties. The demands are especially stringent for superconducting devices as many of the applications are in highly frequency selective, high 'Q' components such as narrow band filters or oscillators where variations of a few percent can have significant impact on the desired device characteristics. Variations in the final performance of a device can arise from many sources. Some obvious factors are the dielectric properties of the substrate or the physical dimensions of the structures such as the etched line widths and substrate thickness tolerances. For HTS the properties of the electrical conductor, the actual superconducting film, are also of importance, affecting the device performance through factors such as the  $T_c$ , the microwave losses and the internal inductance of the superconducting lines. While the quality of commercially available films has improved, the variations routinely found are of the order of several percent. For example, Table 1.a shows the measured  $T_c$ 's from several unprocessed magnetron sputtered films. It is seen that even for pieces from a single wafer (samples 1-4) there is a spread in  $T_c$  of several degrees amounting to ~2% variation. Variations of the same order are found when comparing  $T_c$ 's of wafers produced in the same chamber (samples 5,6). Similar variations are observed in films produced by other growth techniques such as laser ablation, as noted in table 1.b.

The piece to piece variation can be seen in the microwave properties as well. Table 2 and Figure 1 show the  $T_c$ 's and measured Q's from five ring resonators patterned from laser ablated films. Despite the high values and small spread of the  $T_c$ 's exhibited by these films there is a significant spread in the unloaded Q's as well as a variation in the resonant frequency at 77 K. All resonators showed measurable Q's up to 90 K suggesting that any possible  $T_c$  degradation during processing was very small. Since the processing was the same for these samples, it may be suggested that the observed film-to-film variation of the unloaded Q's is determined by the

morphological and structural properties (i.e., grain boundaries, vacancies, surface roughness, etc) of the particular film.

In addition to what may be inherent differences in the HTS film properties, degradations (as measured by  $T_c$ ) were observed for some films as they were processed into circuits. As a means of monitoring this, films typically 2500-3500 Å thick on 20 mil thick LaAlO<sub>3</sub> substrates were obtained from commercial vendors. Films grown both by laser ablation and off-axis magnetron sputtering were investigated. A four-point-probe technique was used to measure the  $T_c$  of the films as received and at intermediate steps as the films were processed into test patterns. After the initial  $T_c$  measurements (no processing involved), the following process steps were investigated:

- (1) Pattern etching:
  - a. Clean sample's surface: 5 min. in acetone in ultrasonic cleaner, followed by 5 min. in ethanol in ultrasonic cleaner and blow dry with nitrogen gas (GN<sub>2</sub>).
  - b. Spin coat positive photoresist (AZ4210); softbake at 90°C for 30 min.
  - c. Expose (1.5 min at 20 mW/cm<sup>2</sup>) and immerse in developer (4:1 H<sub>2</sub>O:AZ400K developer) for 2.5 min. with constant agitation; rinse in deionized (DI) water and blow dry in GN<sub>2</sub>.
  - d. Etch samples:
    - i. In diluted H<sub>3</sub>PO<sub>4</sub> (H<sub>2</sub>O:H<sub>3</sub>PO<sub>4</sub>) 100:1 concentration; cool solution to room temperature; etch for 20 sec.; remove, rinse, and blow dry; re-etch as required in 10 sec. intervals.
    - or:
    - ii. Etch as above in saturated EDTA.
  - e. Strip photoresist:
    - i. Soak in acetone 5 min.
    - ii. soak in methanol 5 min.
  - f. Perform  $T_c$  measurements.
- (2) Contact deposition:
  - a. Spin on positive photoresist (AZ4210); softbake at 70°C for 30 min.
  - b. Expose at 20 mW/cm<sup>2</sup> and soak in chlorobenzene for 10 min. at 23°C; blow dry in GN<sub>2</sub>; bake for 5 min.
  - c. Develop contact pattern (using Shipley CD-30 Developer); Solution temperature 25 ± 1°C; develop for ~2.5 min. with constant agitation; rinse in DI water and blow dry in GN<sub>2</sub>.
  - d. Perform O<sub>2</sub> plasma (50 W rf) cleaning for 30 sec.
  - e. E-beam evaporate: 1000 Å silver followed by 1500 Å of gold.
  - f. Liftoff: soak in acetone, 5 min.; soak in methanol, 5 min.; blow dry in GN<sub>2</sub>.
- (3) Contact anneal:
  - a. Purge tube furnace with 5 SLM of O<sub>2</sub>; insert sample at 200°C; ramp temperature at 20°C/min. to 425°C in 5 SLM O<sub>2</sub>.

- b. Reduce O<sub>2</sub> flow to 1 SLM and hold sample at 425°C for 50 min. Cool slowly to 200°C at 1 SLM O<sub>2</sub>; remove sample from furnace at T ≤ 200°C.
- c. Perform T<sub>c</sub> measurements.

In addition to the above processing steps, the integration of individual HTS-based components into the complete HTSSE-II receiver required some additional processing steps. The first process cured a silver-based epoxy used to attach semiconductor die to the superconducting circuit, this epoxy and heat curing was also separately used to attach the complete HTS circuit to a metal (gold plated KOVAR) sub-carrier. The second process was a vacuum bake-out used to drive off adsorbed moisture immediately prior to the hermetic sealing of the receiver package.

- (4) Epoxy cure cycle treatment:
  - a. Heat in air at 150°C for 1 hr.
  - b. Perform T<sub>c</sub> measurements.
- (5) Vacuum bake-out:
  - a. Heat under vacuum (< 10 mtorr) at 100°C for 24 hr.
  - b. Perform T<sub>c</sub> measurements.

Figure 2 shows a chart of T<sub>c</sub> following the processing steps as observed for laser ablated (LA) and magnetron sputtered (MS) YBCO thin films. For the laser ablated samples the T<sub>c</sub> did not change appreciably as a function of the processing steps, although a small drop in T<sub>c</sub> (less than 1 K) was observed for the sample etched with H<sub>3</sub>PO<sub>4</sub> after the annealing in oxygen. These two samples belonged to the same wafer and their behavior suggest that samples with T<sub>c</sub>'s ≥ 90 K are less sensitive to processing. The sputtered samples exhibited a greater sensitivity to processing and showed a wider variation in T<sub>c</sub> degradation. Samples with similar T<sub>c</sub>'s at step 1 and 2 show different behavior with respect to processing during the remaining processing steps (e.g., MS1 and MS2). It is also apparent that for the magnetron sputtered samples step 3 appears to be a "turning-point" with regards to their T<sub>c</sub> values. The annealing step was further investigated by subjecting unprocessed (i.e. unetched, unmetallized) films to the annealing cycle and monitoring the T<sub>c</sub>. The results of this test are shown in table 1.a and show a great sensitivity and apparent random variability of the sample T<sub>c</sub> to annealing. This behavior was characteristic of many of the magnetron sputtered and, to a lesser extent, of the laser ablated samples examined in our laboratory.

### III. OHMIC CONTACTS

Another area important for HTS applications is the formation of ohmic contacts with low contact resistivity, good adhesion and characteristics which allow the formation of strong wire bonds. In producing the HTSSE II circuits, the strength of wire bonds made to the ohmic contacts were found to be highly dependent on the process used to produce the contacts. Wire bond strengths were determined by conducting pull tests as outlined in MIL-STD-883 and were performed for 0.7 mil gold wire and 1 X 2 mil gold ribbon. In general, the procedure necessary to produce a quality contact depended upon the deposition method used to produce the HTS film,

i.e. depended upon if the film was produced by laser ablation or magnetron sputtering. For magnetron sputtered samples, the following "types" of contacts were studied:

- (1) Type-1:
  - a. E-beam evaporation of 1000 Å of silver followed by 1500 Å of gold.
  - b. Annealing at 425°C in O<sub>2</sub> for 50 min.; cool to room temperature in flowing O<sub>2</sub>.
- (2) Type-2:
  - a. O<sub>2</sub> plasma cleaning, 50 W, 1 min.
  - b. E-beam evaporation of 1000 Å silver followed by 4000 Å of gold.
  - c. Annealing; same as for type 1.
- (3) Type-3:
  - a. O<sub>2</sub> plasma cleaning, 50 W, 1 min.
  - b. E-beam evaporation of 500 Å silver followed by 4000 Å of gold.
  - c. Annealing; same as for types 1 and 2.

Results of the pull tests are shown in Figs. 3 (1 X 2 mil ribbon) and 4 (0.7 mil wire). The results show increasing pull strengths from contact type 1 to type 3, with only type 3 consistently showing strengths which meet MIL-STD-883. The increasing bond strength correlates with the decreasing fraction of silver in the contact metallization. The original rationalization for depositing silver was to lower the contact resistance. However, these tests indicate that the silver has a negative effect on the bond strength.

Similar studies were done with samples deposited by laser ablation to determine the effect of cleaning the HTS surface prior to deposition of the contact metallization. Three processes for cleaning the surface of the contact area were compared: a surface etch using dilute hydrofluoric acid, a surface etch using diluted phosphoric acid, and an oxygen plasma discharge clean.

- (1) Process-1:
  - a. Dip in HF:H<sub>2</sub>O 1:10 for 10 sec.; rinse in DI water and blow dry in GN<sub>2</sub>.
  - b. E-beam evaporate 500 Å of silver followed by 4000 Å of gold.
  - c. Anneal in O<sub>2</sub> at 425°C for 50 min; cool to room temperature in flowing O<sub>2</sub>.
- (2) Process-2:
  - a. Dip in H<sub>3</sub>PO<sub>4</sub>:H<sub>2</sub>O 1:100 for 4 sec.; rinse in DI water and blow dry in GN<sub>2</sub>.
  - b. Same type of metallization and annealing conditions as in process-1.
- (3) Process-3:
  - a. O<sub>2</sub> plasma cleaning, 100 W, 1 min.
  - b. Sputtering, 40 milliamps, 25 sec. (removed approximately 200 Å of YBCO).
  - c. E-beam evaporation of either, (i) 4000 Å of gold or (ii) 500 Å of silver followed by 4000 Å of gold.
  - d. same annealing conditions as in (1) and (2).

Pull tests of 1x2 mil gold ribbon bonds on these samples produced widely different results. Contacts produced by processes (1) and (2) resulted in pull strengths below 2 grams for all bonds. Contacts made using process (3) had better pull strengths as shown in Fig 5. Two observations are made from these tests. First, cleaning of the HTS surface by the wet etchant process was extremely detrimental to the pull strength. (Similar results were observed in early tests on magnetron sputtered films: very poor pull strengths for surfaces cleaned with a phosphoric acid etch). Second, for contacts produced by process 3, the pure gold contacts have better pull strength than the silver-gold ones again illustrating the negative effect of silver on the contact pull strength. A three-point-probe technique was used to measure the contact resistance ( $\rho_c$ ) for these contacts;  $\rho_c=2.8 \times 10^{-6} \Omega\text{-cm}^2$  and  $\rho_c=9.1 \times 10^{-6} \Omega\text{-cm}^2$  were measured for the gold contacts at 10 K and 77 K, respectively. For the silver-gold contacts  $\rho_c=1.3 \times 10^{-6} \Omega\text{-cm}^2$  for both 10 K and 77 K. These values are consistent with those reported by others [6].

Two other contact types were examined during the HTSSE II project. The first was a sputtered contact consisting of 250 nm of silver followed by 250 nm of gold. Initial tests made when the samples arrived in our laboratory showed moderate pull strengths for both 0.7 mil gold wire bonds ( $\geq 2$  g) and for 1 X 2 mil gold ribbon bonds ( $\sim 10\text{-}16$  g). In subsequent tests made after  $\sim 3$  months of storage in a dry box the 0.7 mil wires could not be bonded to the contacts. The 1 X 2 mil ribbons bonded as before and showed similar pull strengths as the initial tests. Auger analysis was performed on these contacts and instead of a layered gold/silver structure the contact metallizations were found to be intermixed and a uniform mixture of gold and silver from the top surface to the HTS interface. Since the 0.7 mil gold wire has a much smaller bonding area, its bond strength is much more sensitive to the surface properties. It is speculated that the silver at the surface of the contact may be oxidized and prevent the formation of a strong bond. The second contact examined was a pure gold in-situ deposited contact fabricated at JPL's Micro-devices Laboratory [5]. The bonding properties of these contacts were very good ( $\sim 18\text{-}20$  g), consistently producing pull strengths far in excess of the MIL-STD minimum values.

## CONCLUSIONS

In this paper we have presented data on some of the processing issues of HTS thin films faced during our work with HTS-based components for the HTSSE-II experiment. It was observed that not only films deposited by different methods but also those deposited by the same technique can show variations in their initial properties (as evaluated by  $T_c$ ). These initial variations between samples broaden as the HTS films are processed and assembled into components with the result that finished devices rarely have identical performance. We studied, also, several contact processing techniques with the intent to identify a process producing low contact resistance as well as good bond strength. The presence of silver in the contact metallization was found to have a negative effect on the bonding properties of the ohmic contacts. Surfaces cleaned with a plasma discharge showed better mechanical properties than those cleaned with either a hydrofluoric or phosphoric acid etch. The best contacts, in terms of bonding strength and repeatability, were pure gold contacts deposited in-situ. In view of the data presented here we believe that despite all the successful HTS-based components demonstrated so far there is still work to be done aimed at improving the uniformity, repeatability and robustness of these materials so that their excellent electrical properties can be fully exploited in real microwave components and systems.

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Table 1.a  
**Tc Variations Before and After Annealing for  
 Unpatterned and Nonmetallized YBCO on  $\text{LaAlO}_3$   
 Thin Films\***

Sample	Wafer	Initial Tc (K)	Intercept <sup>a</sup>	Final Tc (K)	Intercept
1	Wafer 1	87.0	0.163	59	0.306
2	Wafer 1	90.8	0.046	74	0.250
3	Wafer 1	90.8	0.046	85.5	0.224
4	Wafer 1	88.0	0.138	<87	0.250
5	Wafer 2	>89.5	-0.016	>89.7	0.067
6	Wafer 3	>89.5	-0.046	>89.7	0.019

a) Intercept with ordinate axis representing  $(R/R_{\text{max}}) \times 10$ , R=resistance.

\*) Films were deposited by magnetron sputtering.

Table 1.b

**Tc Variation for Unpatterned and Nonmetallized  
Laser Ablated YBCO on LaAlO<sub>3</sub> Thin Films**

Sample	Batch	Tc (K)	Sample	Batch	Tc (K)
1	1	90.47	10	1	88.71
2	1	90.58	11	1	90.42
3	1	90.69	12	1	89.91
4	1	89.41	13	1	90.61
5	1	89.66	14	2	87.67
6	1	89.71	15	2	88.42
7	1	89.35	16	3	88.83
8	1	90.13	17	3	89.69
9	1	90.90	18	3	89.50

Table 2

**Tc and Q Data for Double Sided Laser Ablated  
YBCO on LaAlO<sub>3</sub> Thin Films**

Sample #	Conductor Level	Tc (K)	Q (@77K)	f (GHz)
294	Strip	91.55	5040	8.407
	Ground Plane	90.24		
295	Strip	91.19	3005	8.404
	Ground Plane	91.11		
296	Strip	91.48	1077	8.420
	Ground Plane	91.29		
297	Strip	91.13	2483	8.419
	Ground Plane	90.52		
298	Strip	91.18	2273	8.417
	Ground Plane	90.70		



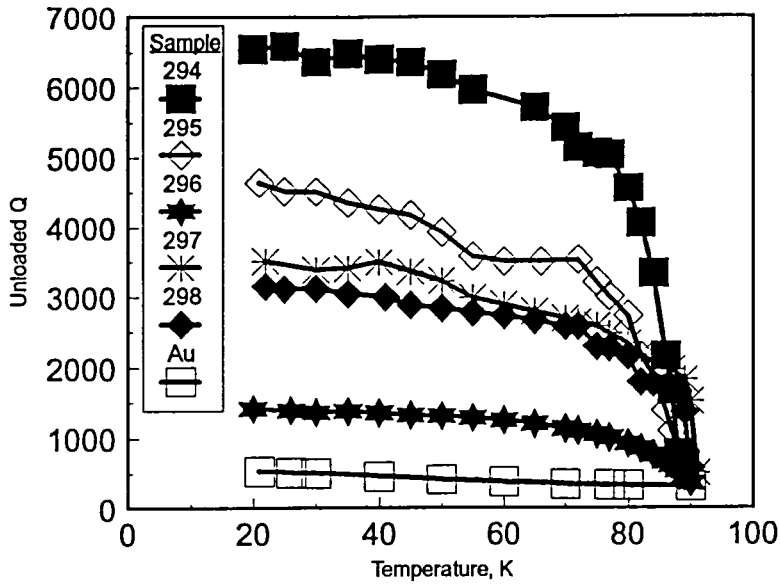


Fig 1. Unloaded quality factor (Q) versus temperature for 8.4 GHz ring resonators etched on laser ablated YBCO on  $\text{LaAlO}_3$ .

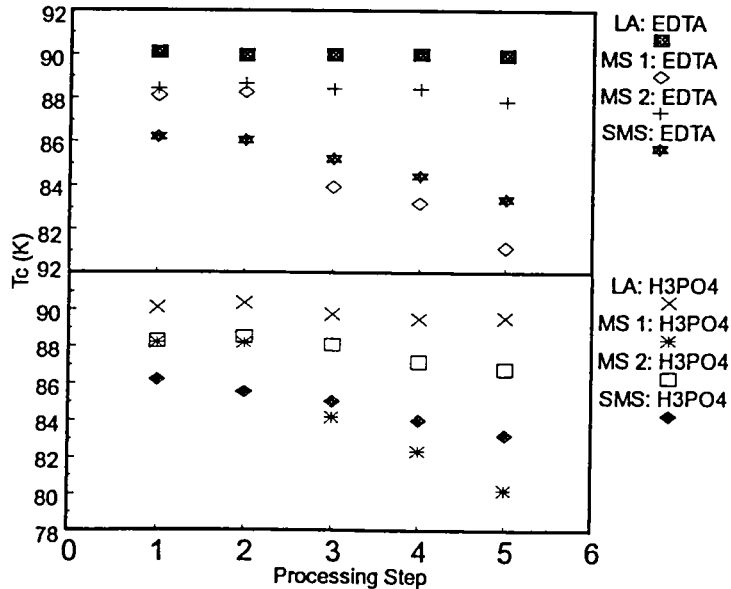


Fig 2.  $T_c$  versus processing steps for laser ablated (LA), magnetron sputtered (MS) and "smooth" magnetron sputtered (SMS) YBCO thin films on  $\text{LaAlO}_3$ . The processing steps are: 1) As received 2) wet etching (either with EDTA or  $\text{H}_3\text{PO}_4$ , 3) contact deposition and annealing, 4) epoxy cure cycle, 5) vacuum bake-out.

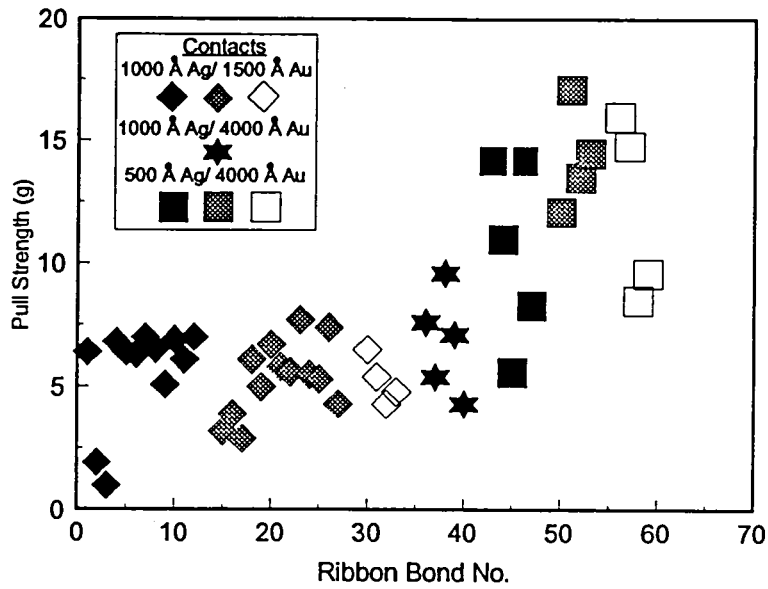


Fig 3. 1x2 mil gold ribbon bond pull strengths for magnetron sputtered samples of YBCO; each shading type corresponds to a different sample.

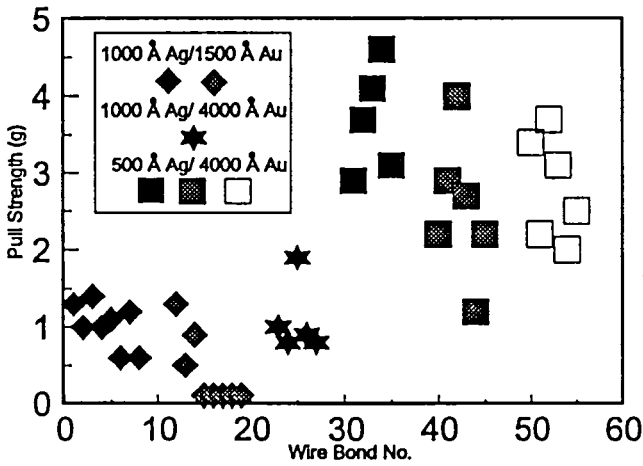


Fig 4. 0.7 mil diameter gold wire bond pull strengths for sputtered YBCO thin films; each shading type corresponds to a different sample.

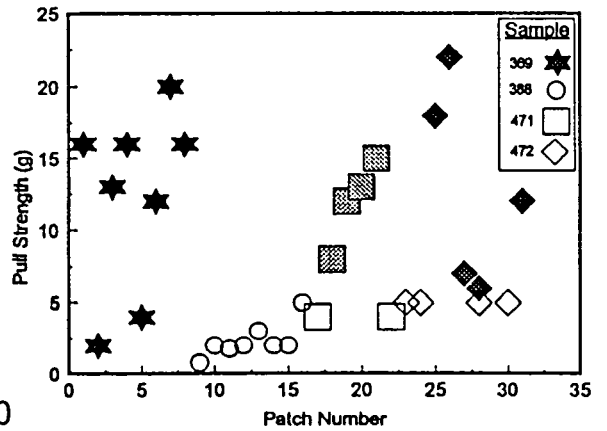


Fig 5. 1x2 mil gold ribbon pull strengths for laser ablated YBCO thin films. Empty symbols are Au/Ag contacts and solid symbols are Au contacts.