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Active

Project #: E-19-X82 Cost share #: Rev #: 0
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Work type : RES
Contract#: N00014-95-C-0090-GT Mod #: Document : CONT
Prime #: N00014-95-C-0090 Contract entity: GTRC

Subprojects ? : N CFDA:
Main project #: PE #:

Project unit: CHEM ENGR Unit code: 02.010.114
Project director(s):
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Sponsor/division names: SUPER CONDUCTOR TECHNOLOGIES / SANTA BARBARA, CA
Sponsor/division codes: 218 / 098

Award period: 950426 to 960130 (performance) 960130 (reports)

Sponsor amount	New this change	Total to date
Contract value	25,000.00	25,000.00
Funded	15,000.00	15,000.00
Cost sharing amount		0.00

Does subcontracting plan apply ?: N

Title: HTS MATERIALS FOR ADVANCED ELECTRONIC APPLICATIONS

PROJECT ADMINISTRATION DATA

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Security class (U,C,S,TS) : U ONR resident rep. is ACO (Y/N): N
Defense priority rating : N/A N/A supplemental sheet
Equipment title vests with: Sponsor GIT X

Administrative comments -
INITIATION OF PROJECT.

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

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NOTICE OF PROJECT CLOSEOUT

Closeout Notice Date 01/21/97

Project No. E-19-X82 Center No. 10/24-6-R8517-0A0

Project Director KOHL P School/Lab CHEM ENGR

Sponsor SUPER CONDUCTOR TECHNOLOGIES/SANTA BARBARA, CA

Contract/Grant No. N00014-95-C-0090-GT Contract Entity GTRC

Prime Contract No. N00014-95-C-0090

Title HTS MATERIALS FOR ADVANCED ELECTRONIC APPLICATIONS

Effective Completion Date 960130 (Performance) 960130 (Reports)

Closeout Actions Required:	Y/N	Date Submitted
Final Invoice or Copy of Final Invoice	Y	960709
Final Report of Inventions and/or Subcontracts	Y	970121
Government Property Inventory & Related Certificate	Y	
Classified Material Certificate	N	
Release and Assignment	Y	
Other	N	

Comments

Subproject Under Main Project No.

Continues Project No.

Distribution Required:

Project Director	Y
Administrative Network Representative	Y
GTRI Accounting/Grants and Contracts	Y
Procurement/Supply Services	Y
Research Property Managment	Y
Research Security Services	N
Reports Coordinator (OCA)	Y
GTRC	Y
Project File	Y
Other	N
	N

NOTE: Final Patent Questionnaire sent to PDPI.

E-19.x.. #1

HTS Materials for Advanced Electronic Applications

Final Report

Contract #N00014-95-C-0090-GT

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1. Statement of Work

This project consists of two tasks.

(i) Polymer passivation: Evaluate possible polymer passivation coatings. These will include both TBCCO and TBCO films for comparison purposes and polymer coating of polyimide, BCB, polyolefin, and epoxies. The superconducting properties and microwave performance will be tested by STI using standard test procedures.

(ii) Inorganic passivation: Investigate simple oxide coatings/ These will include SiO_2 and MgO. A systematic study of deposition conditions and performance of the final device will be performed. The passivation coating material and conditions will be designed so as to bond to the superconductor as completely as possible, keeping the processing temperature, time and moisture as low as possible.

2. Executive Summary

Ten dielectric materials were used to passivate superconducting structures for the purpose of investigating their passivation ability. A variety of processing variables were investigated including the effect of processing time and temperature, and moisture content of the dielectric material, on the microwave properties of the high temperature superconductor (HTS) devices. The changes in microwave performance of a straight line microstrip resonator before and after passivation were quantified by measurements of the loaded and unloaded quality factors for each resonator. It is shown that low moisture content dielectrics which interact as little as possible with the HTS give the best device performance. The effect of temperature is highly nonlinear and does not directly correlate with performance. That is, materials are available which give no degradation in performance if processed at 330^{o}C, whereas others degrade severely at 165^{o}C. (Other properties are however compromised.) This study shows that the degradation of the microwave devices can be minimized by choosing dielectrics which (i) have a low moisture content, (ii) interact as little as possible with the HTS surface, and (iii) can be rapidly processed at relatively low temperatures.

3. Introduction

HTS (high temperature superconductor) circuits offer dramatic performance improvements over conventional technologies for a wide variety of circuit functions. The development of passivation layers which protect the HTS structures from physical and electrical degradation is critical to the deployment of HTS devices. The successful dielectric layer will improve reliability without sacrificing performance and will protect circuits during testing, assembly, and in-service usage. To realize of stable, dielectric passivating material, it is particularly important that the processing conditions required to apply and cure the dielectric material HTS is sensitive to high temperature processing, especially in moist or reducing atmospheres.

4. Experimental

The dielectric materials were selected from those commercially available in electronic packaging. The processing conditions were modified to achieve the time-temperature budget imposed by the HTS material. In general, the polymeric dielectrics were obtained in liquid form and spin cast onto the HTS substrates. For the spin-cast dielectrics, the solutions were dispensed onto the surface of the device in static mode and spun for 30 s at 3000 rotations per minute (RPM). The polymer films were first soft baked to remove the solvent. The films were then hard baked at the times and conditions shown in the results section. The polymers were cured in either a nitrogen filled furnace or on a hot plate under a nitrogen blanket. The plasma deposited inorganic materials were deposited at in a Plasma Therm PECVD reactor under standard conditions (gas flow rates and powers).

Numerous preliminary experiments were run to determine reasonable processing conditions for the detailed experiments shown in the results section. Duplicate samples were run so as to determine the reproducibility of the process.

The test vehicle used to quantify the changes in microwave performance before and after passivation was a straight line microstrip resonator 7 mm long x 150 μm wide. The loaded quality factor was measured for each resonator. The unloaded quality factor (Q_0, a measure of the total loss of the circuit) is calculated from

$$1/Q_0 = 1/2Q_{coup.} + 1/Q_{diel.} + 1/Q_{cond.} + 1/Q_{rad.}$$

where $Q_{coup.}$ is the coupling Q, $Q_{diel.}$ is the conductor Q, and $Q_{rad.}$ is the radiation Q. $Q_{diel.}$ and $Q_{rad.}$ are unknown, but because the geometry is the same for each test, it is assumed that relative changes in calculated unloaded Q are reflections of changes in $Q_{cond.}$

The Q of the TBCCO resonators was measured and used to evaluate the enhancement or degradation of the microwave device properties of the thin film superconductor.

5. Results and Discussion

The passivation materials and processing conditions used in this study are listed in Table 1. The processing conditions were selected so as to minimize the time of exposure to high temperatures, yet still produce acceptable mechanical and electrical properties for the dielectric material.

The Q for each of the resonators was measured before and after processing of the dielectric material. The average percent change in Q is compiled in Table 1. Positive values indicate that the microwave device properties of the superconductor improved during the processing of the dielectric, while negative values indicate that the superconductor degraded as a result of the dielectric processing. It is known that the superconducting properties of YBCO and TBCCO are very sensitive to its oxygen content which can be affected by the moisture and oxygen content of the ambient, particularly at elevated temperatures.

The average changes in the Q factors for the resonators were 9% and 25% for Teflon AF 1601 cured at 330^{o}C and 250^{o}C, respectively. The mechanism for the improvement in the superconductor properties is not understood (beyond the scope of the present study), but improvements are often tied to changes in the oxygen content of the HTS line which could occur during dielectric processing. The original degradation in Q was caused by ion milling damage during resonator fabrication. The reported residual moisture in Teflon AF is very low, 0.01%, so that additional degradation due to reaction with water is minimized. Although quantitative adhesion measurements were not performed, in comparison to the other dielectrics, Teflon AF 1601 did not adhere as well to the resonators and substrates. This set of results shows that higher temperatures can be used without degradation of the HTS (see results at 330 C), although other properties are sacrifices.

Cyclotene 3022 is a thermoset polymer and also has very low residual moisture, 0.1%. The extent of cross linking can be controlled by time and temperature. Five sets of resonators were fabricated and the results are tabulated in Table 1. The lowest temperature process (200^{o}C for 6 min), 39% cured polymer, gave the most favorable results with a 4% improvement in Q. As the temperature was increased to 220^{o}C and 240^{o}C, the average change in Q decreased. Of particular interest is the effect of the adhesion promoter. The Q factors were lower when the adhesion promoter was used for processing at 220^{O}C and 240^{O}C. Thus, the additional chemical bonding to the surface (with adhesion promoter) came at the expense of resonator Q. This supports the observations with Teflon AF, where marginal adhesive forces were correlated with improved Q in all cases. The trade off between time and temperature for the 90% cured Cyclotene 3022 (240^{O}C for 6 min vs. 275^{O}C for 36 sec.) shows that the shorter time process (-22% change in Q vs. -25% change in Q) is most likely better. However, the two results are very similar.

Ultradel 7501, Probimide 293, PI 2540, and PI 2611 were each processed at 200^{O}C for 90 min. The temperature is considerably below that recommended for curing; however, higher temperatures caused considerable degradation of the superconductor. The furnace curing is needed because volatile solvents and/or reaction products are evolved. The average change in the resonators was negative for all four materials, as shown in Table 1. The qualitative observation that exposure of TBCCO to even modest moisture environments is undesirable for long periods of time is substantiated with these materials.

Avatrel dielectric polymer is based on polynorbornene and is known for its excellent adhesion. Although the moisture content is very low and the material can be rapidly processed, the change in Q appears to be due to the high degree of interaction between the polymer and the superconductor. Accuflo 913EL spin-on-glass also resulted in considerable degradation of the superconducting films, in spite of the low temperature and rapid process.

Lastly, several PECVD deposited inorganic dielectrics were investigated, and 0.5 μm of material was deposited for each resonator. In each case the superconductor degraded during processing. It should be noted that the deposition chamber was not equipped with a load lock so that the superconductor was exposed to air at the deposition temperature during pump down, which could contribute to the degradation.

It is anticipated that the failure mode of the resonators involves a change in the oxygen content of the superconductor in the near-surface region. This can occur by reaction with water, oxygen, the dielectric material, or the chemicals used in the processing (adhesion promoters, solvents, etc.). Thus, minimizing the high temperature exposure of the superconductor is important. The interactions between these factors is not quantitatively known. While there is a general trend that lower moisture content is desirable, the processing conditions of the dielectric and surface interactions are clearly important. That is, we found no high-moisture content material to be acceptable, but having low moisture content is not a sufficient condition for preventing degradation.

6. Conclusions

From this work it appears that there are three dielectric processing variables which affect the performance of microwave devices: processing time and temperature, moisture content of the dielectric material, and surface interactions (bonding) with the HTS. Thus, for minimum degradation of device performance, the dielectric should have a low moisture content, interact as little as possible with the HTS surface, and be processed for only a short period at relatively low temperatures. Consequently, the "best" performing dielectrics will have acceptable adhesion (but not in excess) and will most likely not be fully cured.

Passivation Material	Processing Conditions	% Age Change	Average % Change	Unloaded Q (before passivation)	Unloaded Q (after passivation)
ACCUFLO 913EL (SOG)	200C for 2 min	-67		13004	4344
		-72		10644	2999
ACCUFLO 913EL (SOG)	200C for 2 min	-32		12904	8752
		-29		10502	7467
ACCUFLO 913EL (SOG)	200C for 2 min	-12		12927	11433
		-27	-40	11370	8355
BCB (39% cure)	200C for 6 min	5		11101	11673
		-8		10439	9560
BCB (39% cure)	200C for 6 min	12		10904	12184
		6	4	9444	9997
BCB (50% cure) w/ adhesion promotor	220C for 6 min	27		9458	11974
		14		8649	9884
BCB (50% cure) w/ adhesion promotor	220C for 6 min	-13		11580	10110
		-58	-8	9069	3772
BCB (90% cure)	240C for 6 min	-7		11546	10703
		-2		9169	9013
BCB (90% cure)	240C for 6 min	-11		17875	15850
		-7	-4	11948	11170
BCB (90% cure) w/ adhesion promotor	240C for 6 min	-27		16571	12091
		-23	-25	12485	9624
BCB (90% cure) w/ adhesion promotor	275C for 36 sec	-34		14497	9611
		-36		13829	8813
BCB (90% cure) w/ adhesion promotor	275C for 36 sec	-4		12346	11903
		-13	-22	11810	10224
PI - 2540	200C for 90 min	-39		10107	6181
		-37		9323	5871
PI - 2540	200C for 90 min	-37		15482	9812
		-33	-36	12183	8181
TEFLON AF 1601 (6%)	165C 5 min / 330C 12 min	-2		9189	9047
		-9		8329	7613
TEFLON AF 1601 (6%)	165C 5 min / 330C 12 min	40		11149	15650
		3		10580	10874
TEFLON AF 1601 (6%)	165C 5 min / 330C 12 min	20		11544	13910
		1	9	11051	11176
TEFLON AF 1601 (6%)	165C 5 min / 250C 12 min	85		8969	16585
		88		7935	14882

Passivation Material	Processing Conditions	% Age Change	Average % Change	Unloaded Q (before passivation)	Unloaded Q (after passivation)
TEFLON AF 1601 (6%)	165C 5 min / 250C 12 min	29		10629	13730
		8		9310	10057
TEFLON AF 1601 (6%)	165C 5 min / 250C 12 min	-22		14608	11333
		-39	-25	13798	8439
PROBIMIDE 293	200C for 90 min	-35		17335	11258
		-36	-35	15757	10151
ULTRADEL 7501	200C for 90 min	-20		11423	9119
		-23		10912	8420
ULTRADEL 7501	200C for 90 min	-16		12686	10671
		-20	-20	12383	9910
AVATREL 9610	220C for 6 min	-22		13185	10265
		-17		11257	9383
AVATREL 9610	220C for 6 min	-33		14931	9978
		-21		11535	9055
AVATREL 9610	220C for 6 min	-36		11648	7408
		-60	-32	9072	3627
AVATREL 9610	165C 5 min / 250C 60 min	-80	-80	16524	3250
		-80		15491	3074
PI - 2611	200C for 90 min	-60		11124	4410
		-61		8195	3188
PI - 2611	200C for 90 min	-47		15448	8131
		-50		13778	6940
PI - 2611	200C for 90 min	-46		13424	7243
		-41	-51	12064	7064
SiO2	PECVD 0.5mm / 250C	-10		13450	12141
		-18		12626	10410
SiO2	PECVD 0.5mm / 250C	-30		10230	7203
		-31	-22	9006	6183
SiO2	PECVD 0.5mm / 200C	-40		13356	8080
		-41		12550	7451
SiO2	PECVD 0.5mm / 200C	-44		13830	7755
		-43		12571	7152
SiO2	PECVD 0.5mm / 200C	-94		13595	865
		-92	-59	10796	818
Si3N4	PECVD 0.5mm / 250C	-10		13173	11903
		-16	-13	12159	10224