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EFFECTS OF OXIDE FILMS ON SURFACE RESISTANCE AND PEAK FIELDS OF SUPERCONDUCTING NIOBIUM CAVITIES†

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The results of an investigation of residual surface resistance R_{res} and peak rf magnetic fields $\hat{B}$ of large grain niobium cavities in the frequency range of 2 to 4 GHz are presented. It is shown that anodizing the cavities changes the frequency dependence of R_{res} for TM_{01n} modes. Furthermore, comparison is made of Q and breakdown magnetic field $\hat{B}_B$ before and after anodizing. The highest $\hat{B}_B \approx 550$ G were measured in the TM_{010} and TE_{111} modes for cavities heat treated at 1850 °C with no oxide film. The Q's of anodized cavities deteriorate when kept at room temperature even under good vacuum (10^{-7} torr range). Possible mechanisms for this deterioration are suggested.

1. INTRODUCTION

The large decrease of surface resistance when superconducting microwave cavities are cooled below 2 °K has stimulated much research leading to the construction of superconducting electron linacs and rf separators. After considerable work on lead-plated copper cavities at Stanford's High Energy Physics Laboratory (HEPL), at Karlsruhe, at CERN and at Brookhaven, the experiments of Turneaure and Weissmann[1,2] showed that niobium possessed superior properties for practical superconducting microwave devices. With the development of high vacuum firing and chemical polishing procedures at HEPL,[3] niobium became an undisputed candidate for linac and rf separator structures. In fact, two electron linacs are presently under construction, one at HEPL[4] and the other at the University of Illinois.[5] Other linacs and rf separators are in various stages of design or are being studied.

Despite great improvements in the surface resistance of niobium since the early work of Rinderer *et al.*[6] there are still many aspects of superconductivity at microwave frequencies which are not well understood. In particular, the residual surface resistance R_{res} and the peak rf fields which can be maintained on the cavity walls need further study. Equally important is the development of protective coatings on the cavity surfaces to decrease the deterioration of Q factors as a function of time when the cavities are stored in imperfect vacuum.

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Even though the final test of success lies in the realization of a high-Q iris-loaded structure with high deflecting or accelerating gradients, much understanding can be gained by working with simple cavities. Because of our interest in long pulse separated counter beams,[7] we have investigated cylindrical cavities using the manufacturing techniques suitable for the production of iris-loaded structures. The cavities were machined from billets of electron-beam-melted niobium with a grain size of several centimeters and electron beam welded as shown in Fig. 1. The cavities were outgassed, chemically polished and anodized. The results after each treatment can be compared with electroformed and hydroformed cavities in Ref. 8 to which this paper is a sequel.

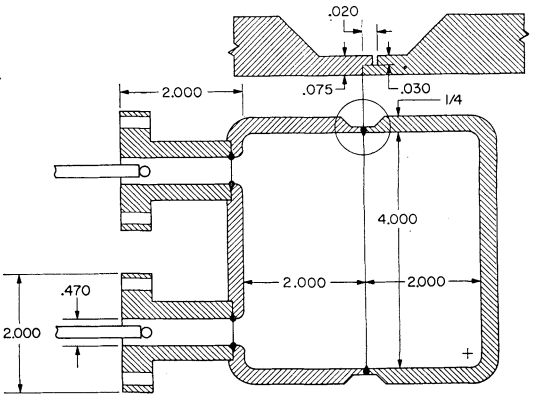

FIG. 1. Niobium cavity with coupling loops. Dimensions are in inches, dots represent electron-beam welds.

2. SURFACE PREPARATION

Since the rf fields react with the material in a very thin layer of only ~ 500 Å the surfaces of the cavities should be smooth and clean. Furthermore, they should maintain this condition during occasional exposure to the atmosphere. Because niobium is a very reactive metal,[9–11] some protective coating should be developed. To accomplish the above objectives, at least partially, one or several of the following surface treatments can be employed.

2.1. *High Temperature Outgassing*

Heating the cavities in a high-vacuum, high-temperature furnace is the single most important treatment for achieving the best results.[3,8] In addition to thermal polishing of the surface, the most objectionable metallic oxide NbO with $T_c = 1.25\,^{\circ}\mathrm{K}$ (Ref. 12) evaporates above 1800 °C. Furthermore, a considerable grain growth which takes place during heat treating enhances the thermal conductivity and results in higher breakdown fields $\hat{B}_B$. It is interesting to note that a prolonged heating (~ 47 hours) of the hydroformed cavity in the Stanford Linear Accelerator Center (SLAC) furnace did not produce any adverse effects except in the TE_{011} mode (Table I). Finally, the outgassing removes the low level limit on the input power observed in the TM_{011} mode.[13] This effect characterized by large frequency pulling and explained as one point multipactoring is treated in some detail in Ref. 14.

2.2. *Chemical Polishing*

Figure 2 depicts schematically the setup for chemical polishing which proceeds as follows: A Teflon valve V_1 from an evacuated stainless-steel container with a polypropylene liner is opened

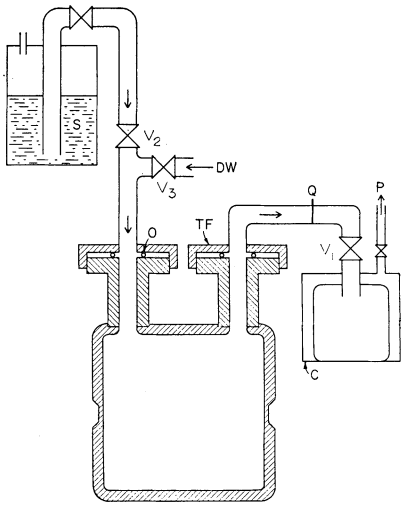

FIG. 2. Apparatus for chemical polishing.

Legend: V=valve, C=stainless-steel container with polypropylene liner, P=vacuum pump, O="O" ring, TF=Teflon flange, Q=quick-disconnect, DW=distilled water, and S=polishing solution.

to bring the cavity to the same pressure, ~ 1 torr. The polishing solution consisting of 40 per cent HF and 60 per cent HNO_3 at 0 to 5 °C is introduced into the evacuated cavity by opening the valve V_2 thereby avoiding air bubbles clinging to the cavity walls. When the flow is established, a quick-disconnect allows the solution to be discarded into a drain. The solution continues to flow until the desired amount of Nb has been removed, ~ 0.001 in. per minute.[15] V_2 is now closed and a copious flow of water is turned on (V_3) to displace the acid without exposing the cavity to air. After thorough rinsing (10 to 15 min) the cavity is filled twice with alcohol. Finally the alcohol is removed by placing the cavity in a vessel which is then evacuated.

TABLE I

Experimental Results of Hydroformed Niobium Cavity

Cavity treatment		°K	TM_{010}	TE_{111}	TM_{011}	TM_{110}	TE_{011}	
Frequency	f		2.26	2.294	2.73	3.607	3.897	GHz
3 h at 1850 °C	Q_0	1.4	1.0	4.8	0.56	1.4	8.5	10^9
	$\hat{B}_B$	1.4	370	350	285	240	350	gauss
47 h at 1850 °C	Q_0	1.4	1.44	8.4	0.89	1.12	3.7	10^9
	$\hat{B}_B$	1.4	321	459	284	313	199	guass

The above procedure produces a smooth surface without pits or orange peel effect, but etches the grain boundaries. All parts in Fig. 2 are made either from polypropylene or Teflon.

2.3. *Electropolishing*

A method which reduces boundary etching was developed by Diepers and Schmidt at Siemens and uses a solution of H_2SO_4+HF at 9 to 15 V. Excellent results were also obtained by electropolishing in this solution at Karlsruhe.[16,17] Due to the geometry of our cavity (Fig. 1) which is not suitable for electropolishing, this method has not been investigated and therefore cannot be compared with chemical polishing which is simpler and more suitable for complex shapes such as the iris-loaded structures of our cavities.

2.4. *Anodic Oxidation*

A rather surprising development in Nb cavities was reported by Martens, Diepers, and Sun,[18] who increased both Q's and breakdown fields $\hat{B}_B$ by coating their cavities with Nb_2O_5. Similar improvements were subsequently reported from Karlsruhe.[16,17]

The anodizing setup used by the author is shown in Fig. 3 and is self-explanatory. Since anodic oxidation is basically the electrolysis of water, one can add any one of a great variety of compounds to increase the electrical conductivity of water.[19,20] The film thickness is determined by the applied voltage and is about 20 Å per volt.[21]

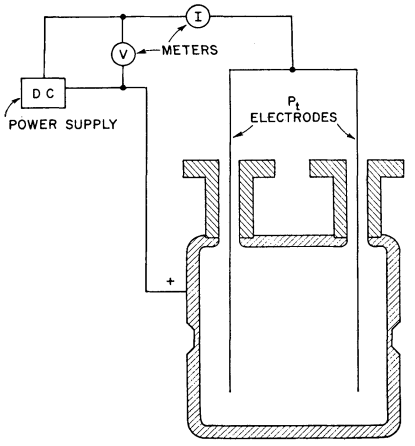

FIG. 3. Anodizing setup.

Our cavities were anodized in 0.2 NH_2SO_4 (Ref. 20) and 20 per cent solution of NH_3 (Ref. 18) at 24 °C to produce films of thickness $d = 200$, 400 and 1000 Å. Investigation with an electron microscope revealed completely amorphous and smooth surfaces. No recrystallization[21,22] was detectable on films formed to 100 V ($\sim$ 2000 Å) with the voltage applied for 10 minutes.

2.5. *Thin Films*

The most promising surface treatment would seem to be the formation of high T_c superconductors by reacting the Nb with tin[23] or nitrogen.[23,24] The question remains how stable these films would be and how much protection for Nb surfaces they will provide. Both films will be studied at Brookhaven in cooperation with M. Strongin and H. Farrell following installation of a high temperature furnace (R. D. Brew and Company) later this summer.

3. SURFACE RESISTANCE

The microwave behavior of superconductors is usually described by the complex surface impedance

$$Z = R+jX,$$

where R is the resistive part related to the ohmic losses in the cavity while X, the reactive part, describes the shift in the resonant frequency. In this paper, the primary interest is in the surface resistance which is determined from Q measurements since R and Q are related by a frequency independent constant G tabulated in Table II. The Q's are measured by a frequency sweep technique[25] in a transmission cavity (Fig. 1) with minimal power input (nW) and coupling coefficients ($<10^{-2}$). The earth's magnetic field is reduced by a μ-metal shield to below 10 mG.

The superconducting surface resistance can be written as

$$R = R_S+R_{\text{res}}$$

where R_S is the strongly temperature dependent part given by the BCS theory and R_{res} is the residual part determined by the condition of the surface layer equalling the penetration depth ($\sim$ 500 Å) of the material.[13]

The frequency dependence of $R_S \propto \omega^{\alpha}$ with

TABLE II

Residual Surface Resistance $T=1.3$ °K

Cavity treatment			TM_{010}	TE_{111}	TM_{011}	TM_{110}	TM_{012}	TE_{011}
G		Ω	302	321	271	481	373	780
Hydroformed	f	GHz	2.26	2.29	2.73	3.6	3.8	3.9
Outgassed at 1850 °C	R_{res}	10^{-7} Ω	1.51	1.13	2.56	2.28	3.7	0.7
Large grain Nb	f	GHz	2.39	2.42	2.88	3.8	4.0	4.12
Outgassed at 1830 °C	R_{res}	10^{-7} Ω	0.63	0.31	1.0	0.56	1.37	0.29
Anodized in 0.2 NH_2SO_4 at 20 V	R_{res}	10^{-7} Ω	0.71	0.75	0.85	0.83	1.07	0.78
Chemically polished and anodized in 0.2 NH_2SO_4 at 20 V	R_{res}	10^{-7} Ω	0.6	0.67	0.69	0.86	0.8	0.39
After 5 days at room temperature and 20 h in air	R_{res}	10^{-7} Ω	1.67	1.23	2.55	2.8	3.22	1.5

$\alpha=(1.8\pm0.2)$ was calculated by Halbritter[26] from the BCS theory and confirmed by experiment[13,17,27] (Fig. 4, curves F and E).

Unlike R_S, the residual resistance, R_{res} is not well understood[13,28] and it is rather surprising that its frequency dependence should be the same as the dependence of R_S, namely $R_{res}\propto\omega^{1.8\pm0.2}$ for both lead-plated copper[29,30] and solid Nb cavities.[17] Even the heat treatment in the high-temperature, high-vacuum furnace does not change the frequency dependence of the TM_{01n} modes as shown in Fig. 4 by the curve A (hydroformed niobium, electron-beam-welded cavity) and curve B (large crystal Nb cavity).

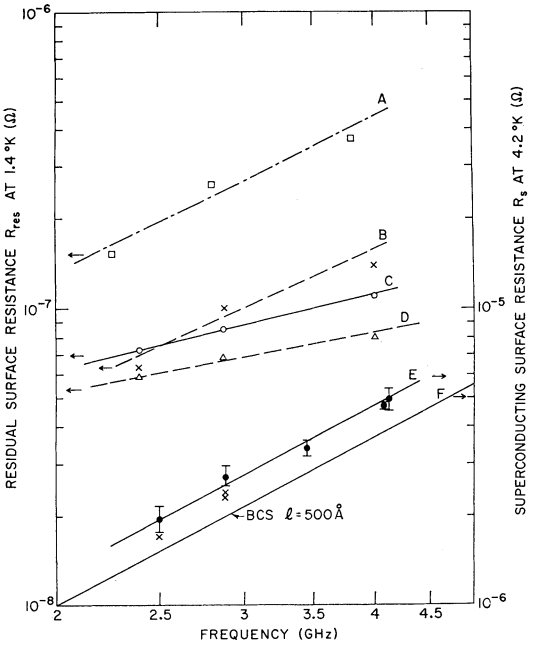

FIG. 4. Frequency dependence of surface resistance of superconducting niobium in the TM_{01n} modes. Residual part R_{res}: $A(\square)$ hydroformed electron-beam-welded cavity outgassed at 1850 °C at SLAC, $B(\times)$ large grain electron-beam-welded cavity outgassed at 1830 °C at HEPL, $C(\bigcirc)$ and $D(\triangle)$ anodized large grain electron-beam-welded cavity. Superconducting part, R_S: Curve F calculated in Ref. 26, Curve E from Ref. 13.

When the outgassed large grain cavity was anodized in 0.2 NH_2SO_4 solution at 20 V the frequency dependence of R_{res} for the TM_{01n} modes became $R_{res}\propto\omega^{0.75\pm0.1}$ indicating that the mechanism of the surface losses has changed (curve C, Fig. 4). Approximately the same dependence was obtained when the cavity was repolished and anodized again in the same solution (curve D, Fig. 4). When the cavities are stored at room temperature, the R_{res} and the exponent α in $R\propto\omega^{\alpha}$ increase. R_{res} for the TE_{111}, TE_{011} and TM_{110} modes are tabulated in Table II for comparison, but their frequency dependence is unknown.

4. HIGH POWER MEASUREMENT

The most important properties a microwave cavity should possess in order to be useful for linear accelerators and rf separators are:

(1) High Q at high power levels.
(2) Capability of maintaining high rf fields on its walls.
(3) Negligible degradation of the above values with time.

The high power measurements are carried out at 1.4 °K by transmission methods with the input loop adjusted for critical coupling while the output loop is coupled very lightly.[8] When large cw powers are fed into the cavity, small changes occur in its resonant frequency due to the pressure changes in the helium dewar, heating of the cavity and the radiation pressure on its walls. The measuring system must therefore be locked in some fashion to the cavity under test.[8,31] Furthermore, since the cavity is a very efficient cryopump while at liquid helium temperatures, a clean vacuum system is very important. The system presently used at Brookhaven contains only copper, stainless steel and glass baked out at 200 °C. Its pressure before cooldown is always in the 10^{-7} torr range.

The best Q's as well as the highest breakdown fields $\hat{B}_B$ were achieved in an electron-beam-welded cavity (Fig. 1) machined from a large grain electron-beam-melted billet and outgassed in the HEPL furnace. Peak fields $\hat{B}$ are calculated from input power measured directly in a critically coupled case.[8] The input power is then increased until a breakdown occurs yielding $\hat{B}_B$. The field enhancement at coupling holes in the TM_{01n} modes is taken into account as in Ref. 8. The results are summarized in Table III and Fig. 5. They can be compared with the small grain hydroformed or electroformed cavities in Ref. 8 or in Table I. Work on similar cavities with indium joints is reported in Ref. 13. The measurements on 'as machined' electron-beam-welded cavities showed a

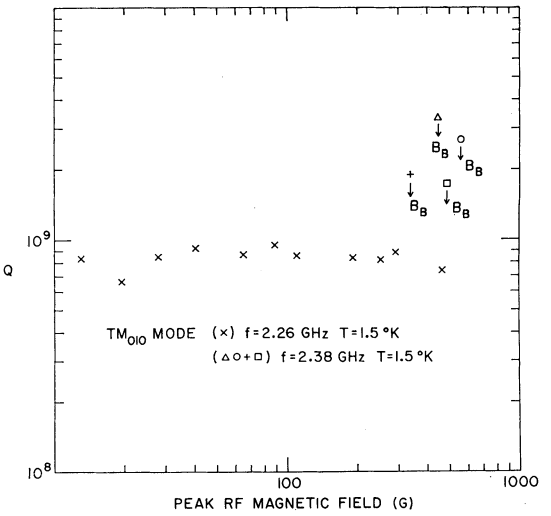

FIG. 5. Q's and breakdown rf magnet fields in a large grain electron-beam-welded cavity for various surface preparations: ○ outgassed at 1850 °C at HEPL, □ anodized in 0.2 NH_2SO_4 (20 V), △ polished and anodized in 0.2 NH_2SO_4 (20 V), +polished and anodized in 20% solution of NH_3 (50 V), × Q versus peak rf magnetic field in a hydroformed cavity.[8]

TABLE III

Experimental results of large grain Niobium cavity

Cavity treatment		°K	TM_{010}	TE_{111}	TM_{011}	TM_{110}	TE_{011}	
frequency		—	2.39	2.42	2.88	3.8	4.12	GHz
As machined	Q_0	1.4	264	922	217	378	2100	10^6
	$\hat{B}_B$	1.4	106	243	70	132	187	gauss
4 h at 1830 °C	Q_0	1.4	2.67	7.11	1.67	3.12	12.8	10^9
at	$\hat{B}$	1.4	357	533	301	269	271	gauss
	$\hat{B}_B$	1.4	554	548	362	358	313	gauss
Anodized in 0.2 NH_2SO_4 at	Q_0	1.4	1.73	6.44	1.73	2.33	8.3	10^9
20 V at	$\hat{B}$	1.4	452	405	43	255	245	gauss
	$\hat{B}_B$	1.4	485	491		313	279	gauss
Chemically polished and	Q_0	1.4	3.35	4.17	1.73	2.6	8.4	10^9
anodized in 0.2 NH_2SO_4 at	$\hat{B}$	1.4	280	408	270	181	251	gauss
at 20 V	$\hat{B}_B$	1.4	442	487	325	264	399	gauss
At room temperature under	Q_0	1.4	1.37	2.62	0.817	1.08	4.79	10^9
vacuum 5 days, 1 day in at	$\hat{B}$	1.4	319	485	204	138	322	gauss
air	$\hat{B}_B$	1.4	379	488	345	276	394	gauss

low field instability at ~ 30 G in the TM_{011} mode, characterized by 3 kHz frequency pulling. The instability was again overcome by operating the cavity at power levels where the breakdown occurs.[13] This mode of operation is commonly called processing. The low Q's are partially due to a rather rough weld with several 'blow-throughs'. After this measurement, the cavity was chemically polished for 30 sec and sent to HEPL. A dramatic improvement occurred in all Q's and $\hat{B}_B$'s with outgassing at 1830 °C for 4 hours (Table III).

The cavity was then removed from the measuring apparatus with a possible exposure to the atmosphere and anodically oxidized in 0.2 HN_2SO_4 (Ref. 21) to 20 V (200 Å). It is conceivable that higher Q's and $\hat{B}_B$'s could be obtained if the cavity were anodized immediately after the removal from the furnace but this measurement must be postponed until we obtain the furnace. Subsequent chemical repolishing and anodizing (Table III) again produced satisfactory results. Processing for 25 minutes in the TE_{111} mode with the peak rf electric field of >10 MV/m and magnetic field of ~ 500 G produced only negligible changes in the measured values. When the cavity was brought to room temperature for a total of 5 days and exposed to the atmosphere of 44 per cent relative humidity, substantial decrease was observed in all Q's while $\hat{B}_B$'s were not seriously affected. (Table III.)

Besides the results listed in Table III we have measured the cavities anodized in a 20 per cent solution of NH_3 (Ref. 32) to 49 V (1000 Å) and in 0.2 NH_2SO_4 to 10 V (200 Å). Also investigated was aging and the effects of processing in the above cavities. It was found that the oxide films produced in 0.2 NH_2SO_4 exhibited both higher Q's and less Q deterioration with time when kept in a good vacuum of 2×10^{-7} torr. Furthermore, all Q's were relatively insensitive to processing in the TE_{111} mode, while the Q's of the cavities which were oxidized in the solution of NH_3 showed a decrease. A steady deterioration in Q's was also observed after each chemical polishing, which is probably due to grain boundary etching, but damage caused by a previous oxide film cannot be excluded. The changes in the breakdown fields, on the other hand, were insignificant in the above measurement.

5. CONCLUSIONS

(1) The comparison of the three manufacturing techniques[8] suitable for the construction of the iris-loaded structures shows that the electron-beam-welded cavities machined from a large grain material yield the best results. In these cavities the improvement factors $I = Q_{Sc}/(Q_{Cu}$ at 20 °C) at high field levels were larger than 10^5 in all modes with the exception of the TM_{011} mode. The highest rf magnetic and electric fields of 554 G and 20 MV/m respectively were obtained in the TM_{010} mode at 2.385 GHz. They are compared with the results for other surface treatments and cavities[8] in Fig. 5. No X-ray radiation was observed at these levels indicating that the breakdown was due to the magnetic field.

(2) A 50-hour-long heat treatment of a hydroformed cavity (grain size ≈ 0.5 cm) in the SLAC high-temperature furnace produced a further enhancement in crystal growth wih only small deepening of the grain boundaries. Since no adverse effects were recorded on Q and $\hat{B}_B$ except in the TE_{011} mode (Table I) we can conclude that thermal etching is negligible.

(3) The frequency dependence of the residual surface resistance $R_{res} \propto \omega^{(1.8\pm0.2)}$ in the TM_{01n} modes for both lead and niobium cavities is rather surprising (Table II and Fig. 4) but in agreement with experiments in other laboratories.[29,30] Due to the narrow bandwidth of the existing microwave measuring equipment (2 to 4 GHz) we cannot conclusively determine the frequency dependence in other modes. There are indications, however, that the exponents might be larger than 1.8 in the TE_{11n} and TE_{01n} modes.

Besides other possible contributions to the residual surface resistance[13,28,33] the same frequency dependence for both residual and superconducting parts suggests the presence of a thin film of NbO on the surface of the cavity.[9] The transition temperature of NbO $T_c \approx 1.25$ °K (Ref. 12) is increased by the proximity effect. When the surfaces are anodized the NbO film is converted to Nb_2O_5 (Ref. 34), a good insulator. The frequency dependence changes to $R_{res} \propto \omega^{0.75\pm0.1}$ which is close to the dependence in the extreme anomalous region[35] $R \propto \omega^{2/3}$. When the oxidized cavities are maintained at room temperature some

Nb_2O_5 at the metal oxide interface changes to NbO, while the extra oxygen diffuses into bulk niobium. Both effects tend to increase the exponent α in $R_{res} \propto \omega^{\alpha}$. One must also keep in mind the increase in the measured Q due to the absorption of rf power given by loss tangent ($\tan \delta$) of Nb_2O_5.

(4) Anodic oxidation produces best results when applied immediately after heat treatment or first chemical polishing. The biggest drawback of the anodized surfaces however lies in Q deterioration with time at room temperature even in good vacuum. Possible causes are: (a) the changes in the oxide itself due to migration of defects resulting in an increased $\tan \delta$,[34] or (b) the reaction of Nb_2O_5 with niobium at their interface,[34] such as formation of NbO and diffusion of oxygen into niobium, which locally decreases T_c of the Nb surface layer.[36] All the above effects tend to degrade all Q's but leave $\hat{B}_B$'s relatively unaffected (Table III). From our experience, thin films (~ 200 Å) formed in a solution with higher electrical conductivity seem to be preferable for complex geometries. A program has been started in cooperation with H. S. Isaacs to develop an optimum anodizing solution and procedure.

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