EFFECT OF VARIOUS GAS MIXTURES ON PLASMA
CLEANED CERAMICS

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Activated gas plasma cleaning efficiencies of various gas mixtures were studied for the preparation of ceramic parts. Differences in the ease of removal of organic residue are shown by Auger analyses for the various gases. A reduction in plasma process time is shown to be possible, and gases are compared with oxygen on the basis of cleaning efficiency. The plasma cleaning processes and changes suggested by the work are discussed.

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SUMMARY

Activated oxygen gas plasma is used to clean ceramic parts. The plasma cleaning efficiencies of various gases were determined and compared with oxygen for five different processing times. Auger analyses of the ceramic parts before and after plasma cleaning indicated the extent to which organic contamination had been removed. Nitrogen, argon, and gas mixtures containing less than 15 percent oxygen showed minimal cleaning potential compared to pure oxygen. However, gas mixtures with oxygen contents of 15 percent or greater showed good cleaning potential and, in particular, dry air was found to be equal to oxygen as a gas for plasma cleaning in all conditions tested.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this work was to determine the effects of various gas mixtures on ceramic part plasma cleaning efficiency. That information would then be the basis for developing a process using a plasma gas with a low concentration of oxygen for production area use.

ACTIVITY

In production, some ceramic parts are machined while potted in paraffin wax. Conventional solvent cleaning of the ceramic was unsatisfactory because an organic residue remained even after cleaning. Since organic residues in the assembly for which these parts are made are thought to be the cause of continued contact loop resistance problems, a better cleaning method was necessary. An activated oxygen gas plasma cleaning process was developed. The process efficiently removes organic contamination from parts. However, it was felt that gas mixtures containing less hazardous amounts of oxygen could still provide satisfactory cleaning using appropriate power levels and processing time.

Plasma Cleaning Process

Plasma is a condition in which a gas can be excited on an atomic level when subjected to an electric field. When excited the gas can form ions, free radicals, or other reactive species. The excited gas, when in contact with organic contamination, can break bonds to form smaller molecules which are swept away by the flowing plasma gas. Under vacuum, the gas can be excited at room temperature and below so that heat buildup or damage to parts from a hot environment is not a problem. Different gases react differently to electric fields and very little is known about the actual mechanisms by which activated gas plasma cleans.

Plasma Process Variables

In activated gas plasma cleaning, the variables are the flow of the gas into the evacuated chamber, the wattage applied to excite the gas, the vacuum level in the chamber, the processing time (time in the plasma) and, in this case, the type of gas used.

The gas flow and vacuum level are mutually dependent within any particular vacuum system; setting one determines the other. The vacuum level for this work was held at 53 Pa absolute pressure which limited the gas flow to approximately 75 cm³/minute for the gases used.

The applied wattage in this case is the RF field generated across the chamber to excite the gas inside. All plasma work was performed with a chamber 152 mm in diameter and 1 m long. The applied wattage used in this work was 50 watts for all gases.

Processing time, the amount of time the samples were actually in the activated gas plasma, was varied from 45 minutes down to 5 minutes, depending on the plasma cleaning ability of the individual gases.

The types of gases used in this work were selected in order to provide a substitute to using pure oxygen in the plasma system. Gases containing approximately 20 percent oxygen or less are acceptable from an explosion hazard standpoint. Mixtures of oxygen and argon containing 5, 10, 15, and 20 percent oxygen were used. Dry air, nitrogen, and argon were also tested individually. A total of eight different gases were tested, including pure oxygen.

The process presently used in cleaning production ceramic parts is 30 minutes in 53 Pa of pure oxygen at 50 watts RF power.

Measurement of Cleanliness

The ceramic parts are heavily contaminated with paraffin wax when received by Bendix. Auger analysis before plasma cleaning was used to determine the extent of contamination in the "as received" condition. Each part was analyzed and a ratio of carbon peak height to oxygen peak height was recorded. Since the ceramic composition is almost entirely aluminum oxide, a decrease in carbon/oxygen ratio on the surface would indicate removal of organic residue; then, Auger analysis after plasma cleaning showed the extent to which the contamination had been removed, and the ratio made it possible to compare different processes.

Analysis of Results

This work was performed to define an optimum plasma cleaning process for ceramic parts with regard to type of gas and shortest optimum process time. Cleaning tests were done for 45, 30, 15, 10, and 5 minute periods. At each process time, gases showing

poor cleaning potential were not tested further, except in the case of nitrogen which gave erratic results. The results of Auger analysis after plasma cleaning are given in Figure 1 and show the averages for each condition tested. The individual values for each ceramic part tested are given in Table 1. Three of each of the two parts were plasma cleaned at each condition evaluated.

Nitrogen and argon gases were shown to have relatively low cleaning potentials based on Auger analysis. Although contaminant removal was accomplished by both gases, the amounts of residue remaining after the longest process times indicate that neither of these gases would be satisfactory for this particular plasma cleaning application. Argon plasma, at longer process times, is presently used extensively at Bendix to clean complex metal assemblies and to treat various surfaces for improved adhesive bonding. Nitrogen and argon are roughly equivalent as plasma cleaning gases; therefore, nitrogen can be considered as a suitable substitute for argon in other plasma applications.

The oxygen/argon gas mixtures containing 5 and 10 percent oxygen demonstrate somewhat better cleaning potentials than nitrogen and argon, but they are still relatively low compared to pure oxygen. It is significant, however, that the 10 percent oxygen/90 percent argon mixture was able to remove all organic contamination from the samples after the longest process time of 45 minutes. This is an indication that even poor cleaning gases are satisfactory if given enough time to do the job. Figure 1 shows the effect of shortening the process time by 15 minutes for these two gas mixtures.

The oxygen/argon gas mixtures containing 15 and 20 percent oxygen indicated a marked increase in plasma cleaning efficiency achieved by a small increase in oxygen content. Both gases were able to clean the sample parts at the present production cleaning process time of 30 minutes, but only marginal cleaning was demonstrated at 15 minutes. The results indicate better cleaning by the 15 percent oxygen mixture at 15 minutes. However, those sample parts had a much lower contaminant concentration prior to cleaning than the parts cleaned in the 20 percent oxygen mixture.

The most efficient plasma cleaning was accomplished when using pure oxygen or when using dry air. Organic contamination was eliminated after 10 minutes and marginal cleaning was demonstrated after only 5 minutes by each gas. The most significant result of this work is that dry air was shown to be equivalent to oxygen as a plasma cleaning gas for this application. It is not known why dry air (composed of approximately 78 percent nitrogen, 21 percent oxygen and 0.5 percent argon plus other gases) is a more efficient plasma cleaning gas than the 20 percent oxygen/80 percent argon mixture.

Figure 1. Extent of Contaminant Removal After Indicated Times

Table 1. Contamination Remaining After Ceramic Parts Are Cleaned With Gas Plasma

Plasma Gas (Percent)	Auger Analysis of Carbon Peak to Oxygen Peak Ratios After Indicated Processing Times				
	45 Minutes (Percent)	30 Minutes (Percent)	15 Minutes (Percent)	10 Minutes (Percent)	5 Minutes (Percent)
Oxygen (100)	0	0	0	0	0
	0	0	0	0	8.0
	0	0	0	0	0
	0	0	0	0	0
	0	0	0	0	0
	0	0	0	0	0
	0*	0	0	0	1.3
Dry Air (100)	0	0	0	0	2.0
	0	0	0	0	0
	0	0	0	0	0
	0	0	0	0	0
	0	0	0	0	0
	0	0	0	0	3.0
	0	0	0	0	0.8
Oxygen (20) Argon (80)		0	6.0		
		0	3.0		
		0	0		
		0	3.0		
		0	0		
		0	0		
		0	2.0		
Oxygen (15) Argon (85)		0	0		
		0	0		
		0	0		
		0	0		
		0	0		
		0	0		
		0	0		
Oxygen (10) Argon (90)	0	0			
	0	5.0			
	0	4.0			
	0	11.0			
	0	0			
	0	0			
	0	3.3			

Table 1 Continued. Contamination Remaining After Ceramic Parts Are Cleaned With Gas Plasma

Plasma Gas (Percent)	Auger Analysis of Carbon Peak to Oxygen Peak Ratios After Indicated Processing Times				
	45 Minutes (Percent)	30 Minutes (Percent)	15 Minutes (Percent)	10 Minutes (Percent)	5 Minutes (Percent)
Oxygen (5)	0	6.0			
Argon (95)	2.0	7.0			
	0	10.0			
	0	0			
	0	0			
	0	7.0			
	0.3	5.0			
Nitrogen (100)	16.0	10.0	16.0		
	3.0	0	15.0		
	20.0	0	13.0		
	3.0	0	6.0		
	4.0	0	4.0		
	3.0	0	4.0		
	8.2	1.7	9.7		
Argon (100)	0	17.0			
	5.0	9.0			
	2.0	9.0			
	0	5.0			
	0	4.0			
	3.0	8.0			
	1.7	8.7			

*Values below sum lines indicate averages.

Of the eight gases tested, nitrogen and argon were shown to be too low in plasma cleaning potential to be satisfactory in this application. The oxygen/argon gas mixtures with small amounts of oxygen demonstrated an ability to remove organic contamination, although they were not as efficient plasma cleaning gases as pure oxygen. Dry air, with an oxygen content of approximately 21 percent, was shown to be as efficient as pure oxygen in removing organic contamination from the ceramics. It should be noted that in this work only six ceramic parts were plasma cleaned at a time, as opposed to approximately 350 parts in production cleaning.

Therefore, the cleaning time used in this work might have to be altered for a fully loaded plasma chamber in order to achieve complete contaminant removal. However, the relative plasma cleaning efficiencies of the gases tested would not be changed.

ACCOMPLISHMENTS

The effects of various gas mixtures on ceramic part plasma cleaning efficiency have been determined for eight different gases. Plasma cleaning gases having a low concentration of oxygen (15 to 20 percent) are able to remove organic residues from the ceramics under current production process conditions. Of these gases, dry air is the most satisfactory substitute for oxygen as a plasma cleaning gas.

FUTURE WORK

In order to establish production cleaning processes using the information from this project, a minor amount of testing would be required using holding fixtures and a fully loaded plasma chamber.

The effect of low oxygen concentration gases on the plasma cleaning efficiency of various metal parts and assemblies should now be determined. Considerable improvement over present production cleaning should be possible without deleterious effects on the parts.

The use of sensitive electronic parts and assemblies continues to increase at Bendix. In an effort to improve reliability by contaminant removal, the effects of activated gas plasma on these sensitive parts should be determined by electrical testing.

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