Evaluating the Effectiveness of Solvent Free Removal of Waxes

1.0: Purpose

The mounting of specimens for specimen preparation is a common practice in virtually all metallographic, optical, and electron microscopy applications. In most cases the specimens require some type of polishing technique which necessitates the use of a temporary adhesive to fix the specimen during preparation. The use of low melting point wax is the most common technique, involving the heating of the specimen to a temperature at or near the melting point of the wax being used. The specimen is then affixed to a block or mount of some type, followed by a series of grinding and polishing steps to achieve the desired section and surface finish. Typically the specimens are reheated and then cleaned in a solvent such as acetone or methanol. However, with certain types of specimens such as polymers, plastics, and some types of circuit boards, solvent cleaning cannot be used. For these types of specimens a cleaning solution is required which will not attack and dissolve the specimen, or an alternative method for mounting the samples is required. In this experiment, a new wax removal technique will be tested. A compound which is soluble in water and commonly used for semiconductor wafer cleaning will be tested to evaluate the effectiveness of removing wax from a specimen. Different materials will be evaluated to ensure that the wax removal (or lack thereof) is uniform with most material systems. Comparing the new water cleaning method with traditional solvent cleaning will also be examined.

2.0: Experiments and Procedures

Four different specimen types were tested in this experiment. Samples of silicon (Si), quartz, brass and printed circuit board were all heated to an approximate temperature of 130°C and then a small amount (~ 2 mm^2) of wax was applied to the surface and allowed to cool. Once the specimen had cooled to room temperature, they were placed into a bath of the water soluble cleaner (~ 20 ml) and ultrasonically cleaned for 5 minutes. The cleaner was set temperatures varying from 65°C up to 100°C prior to cleaning of the specimens. Three different ratios of cleaner were tested to determine any possible anomalies in concentration. Once the specimens were cleaned, they were each rinsed by dipping into a beaker of water 3 times and then dried on filter paper.

Four different wax types were tested in this experiment. Below is a table illustrating the properties and types of waxes used for testing.

Designation	Wax Type	**Melting Point (°C)**	**Strength**	**Hardness**	**Solubility**
1	MWH 135	135	High	Very Hard	Acetone
2	MWH 080	80	High	Hard	Ethanol, acetone
3	MWM 070	70	Moderate	Moderate	Warm soapy water
4	MWS 052	52	Moderate	Soft	Perchloroethylene

The wax samples were applied to each specimen type, with a total of 16 samples cleaned. The same process was done using acetone as the primary cleaner, followed by a water rinse as described above.

3.0: Results

Following the cleaning tests, a large data set was collected which showed the results of all of the testing done. Each aspect of the experiments will be discussed in detail below. The charts can be evaluated at the end of this report.

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Table 1

Table 1 is the data collected for the 3.5 to 1 mixing ratio (water to powder) of the WaxStrip solution. This is the smallest mixing ratio tested, making it the most concentrated solution used in the cleaning process. The solution was heated to a temperature of 100°C with a sonication bath temperature of 30°C. Each of the samples were sonicated for 5 minutes and then the surface was examined optically to help determine the amount of wax removal obtained by the cleaning. From the chart it can be seen that for samples contaminated with wax #1 the removal was virtually non existent. However, for the other three waxes, #2, 3, and 4, cleaning was very efficient. The variation in cleaning compared to the various sample types is minimal, with all four different samples cleaned equally, depending upon the mounting wax they were contaminated with.

Table 2

Table 2 is the data collected for the 5.3 to 1 mixing ratio of the WaxStrip solution. This is an in between mixing ratio and is less concentrated than the one used in Table 1. The solution again was heated to a temperature of 100°C with a sonication bath temperature of 30°C. Again, as with the results in Table 1, mounting wax #1 was not removed efficiently as compared with the other three waxes. However, there was some removal of wax #1 in the brass and silicon samples observed. The efficiency of the cleaner to remove the other three types of wax seems to be quite high.

Table 3

Table 3 shows the data collected for a 10 to 1 mixing ratio of the WaxStrip solution. The solution was at a temperature of 100°C with a sonication bath temperature of 30°C. The results were virtually identical to those obtained with the 5.3 to 1 mixing ratio, with slight improvement in the removal of wax #1 on the brass and silicon samples. All other waxes were removed relatively easily.

Table 4

Table 4 shows data collected for a cleaning solution at a 10 to 1 mixing ratio, which was observed to be the optimum mixing ratio from the previous experiments, The cleaning solution temperature was reduced to 80°C and the sonication bath temperature was kept constant at 30°C. No significant change in cleaning characteristics were observed with those of Table 3.

Table 5

Table 5 shows the same experiment as Table 4 with a cleaning solution temperature of 70°C and a sonicating bath temperature of 70°C as well. In this case, all four wax types and sample types were cleaned with equal efficiency. The sonicating bath temperature seems to have had a positive effect on the outcome of the cleaning solutions.

Solvent Cleaning

Some experiments were run using acetone cleaning as a pre cleaning step prior to the WaxStrip cleaning. These were shown to be effective in removing wax residue, although the benefit of solvent free cleaning is lost using this method. A slight mount of residue was seen on the silicon samples which was not present from solvent free cleaning, but is unclear as to whether or not this is due to the solvent step or not. Further experimentation may be needed to evaluate this further.

4.0: Conclusions

Based on the experiments and data collected, the use of a solvent free cleaning solution can be quite useful in removal of most mounting waxes. Without the need for solvent cleaning, specimens which are normally affected by solvents can be cleaned as well. The most critical parameters which affect the outcome of cleaned specimens is the mixing ratio of water to WaxStrip powder; cleaning solution temperature and sonicating bath temperature. The optimum conditions are to keep the mixing ratio at 10:1, keep the sonicating bath and the cleaning solution temperature both the same. All of these combined produce well cleaned specimens.

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TABLE 1

Cleaner Ratio:	3.5 to 1
Cleaner Temp:	100 degrees Celsius
Sonication Time:	5 minutes
Bath Temp:	30 degrees Celsius
Sample	Result
1A	No effect
1B	No effect
1C	No effect
1D	No effect
2A	85 % removal
2B	99 % removal
2C	100 % removal
2D	100 % removal
3A	100 % removal
3B	100 % removal
3C	100 % removal
3D	100 % removal
4A	100 % removal
4B	100 % removal
4C	100 % removal
4D	100 % removal

TABLE 2

Cleaner Ratio:	5.3 to 1
Cleaner Temp:	100 degrees Celsius
Sonication Time:	5 minutes
Bath Temp:	30 degrees Celsius
Sample	Result
1A	No effect
1B	No effect
1C	10 % removal
1D	35 % removal
2A	100% removal
2B	100% removal
2C	95% removal
2D	100 % removal
3A	100 % removal
3B	100 % removal
3C	100 % removal
3D	100 % removal
4A	100 % removal
4B	100 % removal
4C	100 % removal
4D	100 % removal

TABLE 3

Cleaner Ratio:	10 to 1
Cleaner Temp:	100 degrees Celsius
Sonication Time:	5 minutes
Bath Temp:	30 degrees Celsius
Sample	Result
1A	No effect
1B	No effect
1C	75% removed
1D	100% removed
2A	100% removal
2B	100% removal
2C	100% removal
2D	100% removal
3A	100% removal
3B	100% removal
3C	100% removal
3D	100% removal
4A	100% removal
4B	100% removal
4C	100% removal
4D	100% removal

TABLE 4

Cleaner Ratio:	10 to 1
Cleaner Temp:	80 degrees Celsius
Sonication Time:	5 minutes
Bath Temp:	30 degrees Celsius
Sample	Result
1A	85% removal
1B	100% removal
1C	80% removal
1D	100% removal
2A	100% removal
2B	100% removal
2C	100% removal
2D	100% removal
3A	100% removal
3B	100% removal
3C	100% removal
3D	100% removal
4A	100% removal
4B	100% removal
4C	100% removal
4D	100% removal

TABLE 5

Cleaner Ratio:	10 to 1
Cleaner Temp:	70 degrees Celsius
Sonication Time:	5 minutes
Bath Temp:	70 degrees Celsius
Sample	Result
1A	100% removal
1B	100% removal
1C	100% removal
1D	100% removal
2A	100% removal
2B	100% removal
2C	100% removal
2D	100% removal
3A	100% removal
3B	100% removal
3C	100% removal
3D	100% removal
4A	100% removal
4B	100% removal
4C	100% removal
4D	100% removal

LEGEND	
Sample Designation	Wax Designation
A = PC Board	1 = MWH 135
B = Quartz	2 = MWH 080
C = Brass	3 = MWM 070
D = Silicon	4 = MWS 052

WS1

WaxStrip™

Instructions for Use and Application Note #31

WaxStrip™ is an enviromentally safe powder compound prepared primarily for the removal of mounting waxes in semiconductor processing. WaxStrip™ is compatible for use on silicon, glass, iron, aluminum, zinc, brass, copper, magnesium and other materials. It is a water soluble powder that is ideal for the removal of mounting waxes in semiconductor processing

WS1 Mixing Instructions:

Use: Add 6oz wax strip remover per gallon of water. Heat solution to 50-70°C. Immerse parts. Agitation of parts helps the removal process. Rinse thoroughly.

Caution: Can cause burns. Avoid contact with skin, eyes and clothing. Use goggles or face shield. In case of contact flush well with water for 15 minutes. If swallowed DO NOT induce vomiting. Give 1 ounce vinegar in equal parts of water. Call a physician. In case of leakage, flush well with water.