DELAMINATION STUDIES IN Cu-ULTRA LOW-k STACK

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Abstract:

One of the major challenges with ultra low-k materials is to eliminate delamination during the chemical mechanical planarization process due to their poor mechanical integrity. Two patterned Cu samples containing different ultra low-k films (A and B) having k values <2.0 have been investigated. 2-cm^2 coupons of the samples were polished on a bench-top polisher with either upper oscillation or upper rotation setup. Experiments were performed at different machine parameters with alumina based Cu slurry and IC 1000 polishing pad. In every set of testing of A and B samples, a similar patterned Cu sample with TEOS as interlayer dielectric had been used for a direct comparison. Both the dynamic coefficient of friction (COF) and energy of contact high-frequency acoustic emission (AE) were continuously monitored during the tests. COF is found to be less for TEOS sample for down force 3 PSI or less whereas AE shows very high values with higher noise for low-k samples at all conditions. As all Cu removes, AE signals for TEOS and low-k films are similar. Monitoring AE signal along with COF data could be used as an in situ metrology tool for monitoring of Cu damascene process with ultra low-k materials.

Introduction:

The constant push towards sub-micron miniaturization of the device dimensions, increased density of devices, and faster processing power has led to the development of new interconnect technologies that use Copper (Cu) and ultra low-*k (k<22)* polymer based dielectrics [1]. The dielectric constant of the interlayer dielectric films generates high parasitic capacitance, which leads to increased signal propagation delay-time and cross-talk. In the 50 nm node, the International Technology Roadmap for Semiconductors (ITRS) predicts a need for dielectrics with a bulk *k* value of <1.3 and effective *k* value <1.5 for the dielectric stack. The incorporation of Cu as a replacement for previously used conducting interconnect material, Aluminum (and Aluminum alloys), has further reduced the resistance of the metal interconnects and improved the performance of the ICs [2]. Mechanical characterization of low dielectric constant (low-k) materials has shown that lower *k* typically also means lower elastic modulus and hardness [3]. Due to lower mechanical strength, reduced cohesive strength and lack of compatibility with other interconnect materials, major challenges are involved in chemical mechanical polishing (CMP) of the Cu and ultra low-k materials stack [4].

In this study we have investigated the polishing behavior of patterned copper samples with different ultra low-k materials underneath. Both coefficient of friction (COF) and acoustic emission (AE) were continuously monitored during the tests and they were analyzed in order to characterize the delamination behavior of the ultra low-k materials. Optical and scanning electron microscopy was used to investigate the polished surface. An attempt has been made to find the mechanisms of delamination due to the effects of machine parameters and consumables. Correlation of mechanical properties the materials on the delamination behavior of the ultra low-k materials will also be discussed in the paper. Objective of this research is observation and detection of the nature of delamination on different patterned samples during polishing under different conditions.

Experimental:

The real time CMP process has been simulated on a Bench-top CMP Tribometer. Fig. 1 shows the schematic of the CMP process with a oscillatory upper specimen movement adopted in the laboratory. The details of the CMP tester and its effectiveness in the CMP process have been previously discussed [5]. Three different types of samples have been used in this study:

1. Patterned Cu samples with low-k A (A) (k~<2)
2. Patterned Cu samples with low-k B (B) (k~2.0)
3. Patterned Cu samples with SiO2 (TEOS) (k~4.0)

Later in the text the samples will be discussed as A, B and TEOS respectively for low-k A, low-k B and SiO_2 films. Small coupon (2cm X 2cm) of samples were used to polish on either 6" or 9.5" pad coupon at different rotation of platen (0.2 to 1.5 m/s) and down force (1-10 PSI). Tests were done either in a machine with active upper rotation or in a machine with no upper rotation. For no upper rotation the contact interface was a 6" polishing pad coupon as the lower specimen and a wafer coupon as the upper specimen. Upper specimen was allowed to oscillate back and forth by 5 mm (47.5-52.5 mm) with a speed of 1 mm/s. Polishing slurry was fed into the interface continuously with the rate of 55 ml/min. Details of the process conditions and consumables are presented in Table 1. There were two stages of polishing. In first stage applied force gets settled and polishing occurs for 7 sec and then second stage starts for 300 sec. In some occasions, tests were stopped before 300 sec due to high vibration and sample holder rotation.

Table 1. Polishing conditions of sample A, B and TEOS on the machine with no upper rotation. Slider movement for these experiments were 50±2.5 with a velocity 1 mm/s. Slurry flow was fixed at 55 ml/min. Polishing pad was a perforated ICl000/SubalV while alumina based Cu selective slurry was fed continuously at the center of the pad. Samples 1-5 were polished with relative velocity 0.8 m/s for different PSI while samples 6-10 were polished with 3 PSI at different relative velocities.

Coupon No.	Coatings	PSI	Time (sec)	Coupon No.	Coatings	RPM	Time (sec)
001	A	0.5	312	006r	A	250	312
001	B	0.5	312	006	B	250	312
001	TEOS	0.5	312	006	TEOS	250	312
002	A	1	312	007	A	200	312
002	B	1	211	007	B	200	312
002	TEOS	1	312	007	TEOS	200	312
003	A	2	312	008	A	150	312
003	B	2	312	008	B	150	312
003	TEOS	2	312	008	TEOS	150	312
004	A	4	312	009	A	100	312
004	B	4	312	009	B	100	312
004	TEOS	4	312	009	TEOS	100	312
005	A	6	312	010	A	50	312
005	B	6	312	010	B	50	312
005	TEOS	6	312	010	TEOS	50	312

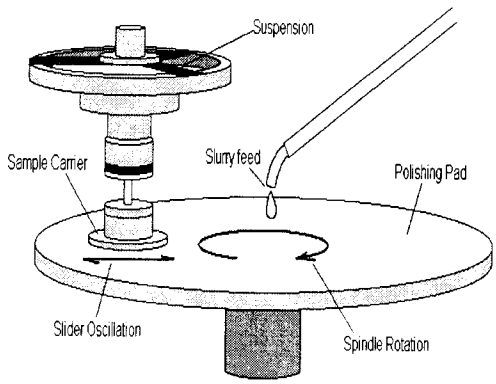


Fig. 1 Schematic of the CMP process

For active upper rotation the contact interface was a 9.5" polishing pad coupon as the lower specimen and a sample coupon was the lower specimen. Experiments with active upper rotation were performed at CETR, Inc., Dell Avenue, CA facilities. Details of the polishing conditions and consumables are given in Table 2. Both the dynamic coefficient of friction (COF) and energy of contact high-frequency acoustic emission (AE) were continuously monitored during the test. Optical and scanning electron microscopy was used to investigate the polished surface.

Table 2. Polishing conditions of sample A, B and TEOS on the machine with active upper rotation. Slider movement for these experiment was 62.5+2.5 with a velocity 1 mm/s. Slurry flow was fixed at 30 ml/min. Polishing pad was a K-grooved ICl000/SubaIV while alumina based Cu selective slurry was feed continuously at the center of the pad.

Coupon #	Coating	Conditions		Time (sec)
		PSI	RPM	
ACL-6	A	7	120/110	304
ACL-2	A	7	120/110	34
ACL-7	A	4	120/110	34
ACL-8	A	4	120/-	34
ACL-3	A	10	60/50	304
BCL-4	B	7	120/110	304
BCL-5	B	7	120/110	34
BCL-9	B	4	120/110	34
BCL-6	B	4	120/-	34
BCL-2	B	10	60/50	304
TC-12	TEOS	7	120/110	304
TC-13	TEOS	7	120/110	34
TC-5	TEOS	4	120/110	34
TC-7	TEOS	4	120/-	34

Results and Discussion:

During polishing AE signal and COF data were recorded and analyzed at different stages of polishing. Details of the results are summarized in Table 3-6. Variation of COF with RPM and PSI are shown in Fig. 2. It can be seen from Table 3 and Table 4 and Fig. 2 that COF for all three different types

of samples decreases with the increase of platen velocity (RPM). COF also has a downward trend with the increase of PSI. TEOS sample has lowest COF for most of the conditions used. Sample B has a trend of a

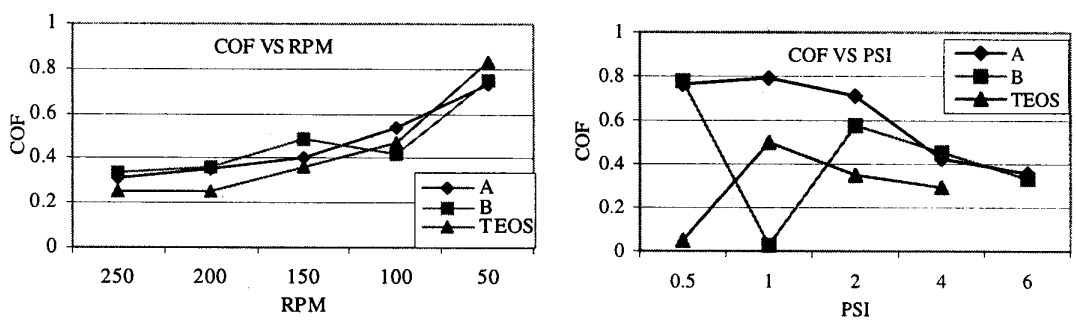
Table 3. Values of COF at different conditions and at different stages of polishing. Polishing was performed at constant RPM (0.8m/s) with varying PSI.

PSI	A (001-005)			B (001-005)			T (001-005)		
	a	b	c	a	b	c	a	b	c
0.5	0.4712	0.7631	0.3551	0.5324	0.7790	0.4240	—	—	—
1.0	0.4919	0.7929	0.3569	0.0255	0.0267	0.0205	0.0379	0.0479	0.0337
2.0	0.5761	0.7118	0.5057	0.3455	0.5781	0.2550	0.3666	0.4994	0.3058
4.0	0.2569	0.4257	0.1928	0.2499	0.4555	0.1708	0.2691	0.3496	0.2364
6.0	0.2240	0.3583	0.1744	0.2228	0.3318	0.1938	0.2557	0.2936	0.2356

*a = Full time b = 10 - 50 sec c = 150 - 250 sec For B002 total time of polishing is 211 sec. And range c is 150-190sec.

Table 4. Values of COF at different conditions and at different stages of polishing. Polishing was performed at constant PSI (3 PSI) with varying RPM.

RPM	A(006r-010)			B (006-010)			T (006-010)		
	a	b	c	a	b	c	a	b	c
250	0.2035	0.3082	0.1642	0.2223	0.3373	0.1793	0.2030	0.2558	0.1813
200	0.2160	0.3490	0.1649	0.2242	0.3579	0.1774	0.1721	0.2536	0.1417
150	0.2445	0.4074	0.1774	0.2910	0.4912	0.2218	0.2573	0.3607	0.2193
100	0.3364	0.5397	0.2434	0.3153	0.4167	0.2853	0.2931	0.4707	0.2186
50	0.6160	0.7336	0.5791	0.6221	0.7511	0.5364	0.6152	0.8284	0.5816



(a) (b)

Fig. 2. (a) Variation of COF at different RPM. Values were taken in the time interval of 10-50 sec. of polishing from the second stage and (b) variation of COF at different PSI. Values were taken in the time interval of 10-50 sec. of polishing from the second stage. Small values (below 0.05) for B and T samples are due to hydroplaning.

little higher COF than sample A. All three samples have a similar pattern and a top Cu layer, which is being polished during the process. In principle, COF should be same for all the samples at the beginning of the polishing as polishing is taking place only on the top surface. As Cu is removed (even partially), the barrier layer will appear and then the underneath ultra low-k ILD film gets exposed to the CMP environment. It is also noticed that at the end of 300 sec of polishing when Cu is removed partially (Fig.3)

COF is lower for the TEOS samples. This is may be due to the fact that sample A and B undergoes leading edge delamination and exposure of ILD to the CMP environment, whereas no delamination is taking place for TEOS sample. Decrease of COF for all samples with time is due to the fact that the surface of Cu film gets smoother as polishing progresses. It can be seen from Fig. 2 that variation of COF with higher PSI for three different samples is more than the samples polished with different RPM at lower PSI (3 PSI). Recording the COF values below 0.1 is may be due to the effect of hydroplaning.

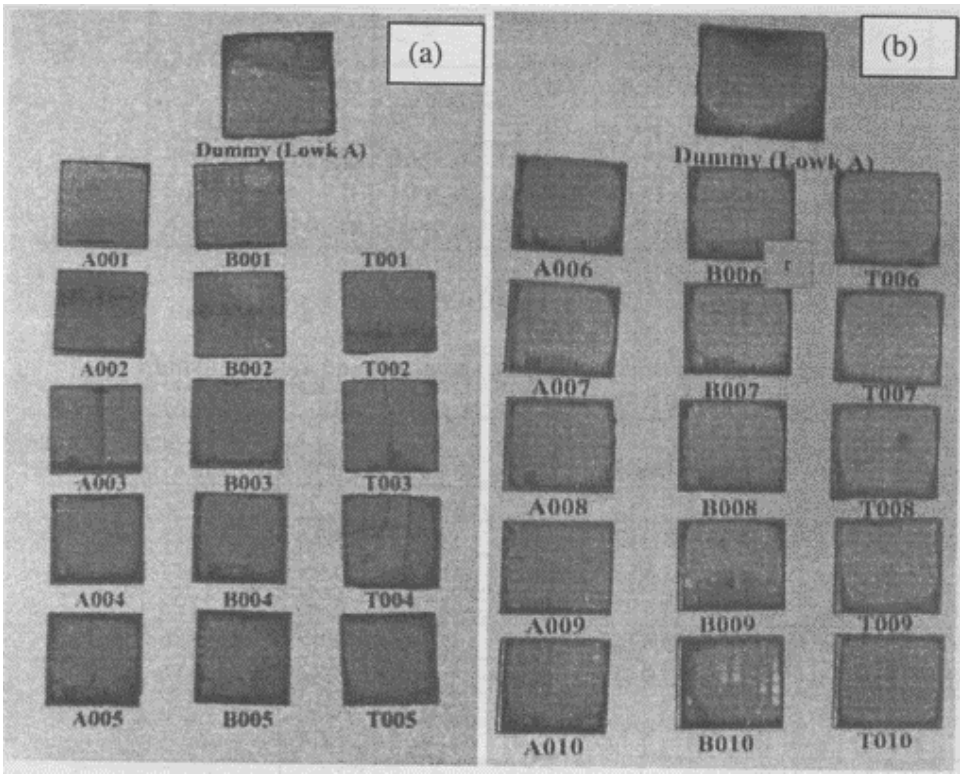


Fig. 3. (a) Picture of sample coupon after polishing at constant RPM with varying PSI and (b) Picture of sample coupon after polishing at constant PSI with varying RPM.

It can be seen from Table 5 and Table 6 that AE signal for all the samples increases with the increase of both RPM and PSI. AE signal represents the intensity of polishing and as the relative velocity and PSI increases, higher interactions among the pad, slurry particles and film's surface occur and hence

Table 5. Values of AE at different conditions and at different stages of polishing. Polishing was performed at constant RPM (0.8m/s) with varying PSI.

PSI	A (001-005)			B (001-005)			T (001-005)		
	a	b	c	a	b	c	a	b	c
0.5	1.99	3.36	1.4638	2.3	3.62	1.7643	-	-	-
1.0	4.02	7.92	2.3973	0.3592	0.4075	0.1795	0.3605	0.62	0.2643
2.0	3.92	8.09	2.1462	2.52	6.12	1.1048	0.7276	1.42	0.4096
4.0	2.53	6.19	1.1019	2.13	5.5	0.8406	1.65	3.47	0.7732
6.0	2.90	6.93	1.3881	2.68	6.53	1.4313	1.24	1.90	0.7829

the higher AE signals. A typical AE signal curve of three different types of samples polished with Cu slurry without upper sample carriage rotation are shown in Fig. 4. It can be noticed that at the beginning

of the polishing AE signal is more for sample A and B than TEOS but at the later stage of polishing the variation is less.

Table 6. Values of AE at different conditions and at different stages of polishing. Polishing was performed at constant PSI (3 PSI) with varying RPM.

RPM	A (006r-010)			B (006-010)			T (006-010)		
	a	b	c	a	b	c	a	b	c
250	4.4327	9.3809	2.4333	4.1322	9.5313	2.0705	2.7225	5.7524	1.4033
200	3.4157	7.7525	1.6832	0.8943	2.2469	0.4079	1.3164	2.98338	0.6949
150	2.6048	5.9442	1.2232	1.9268	5.1737	0.7007	1.0107	2.0954	0.5070
100	1.3040	2.5800	0.7395	0.9005	1.8870	0.5296	0.7218	1.6250	0.3595
50	1.2795	1.4398	1.1135	1.5099	1.3109	1.8333	1.4822	2.1754	1.2703

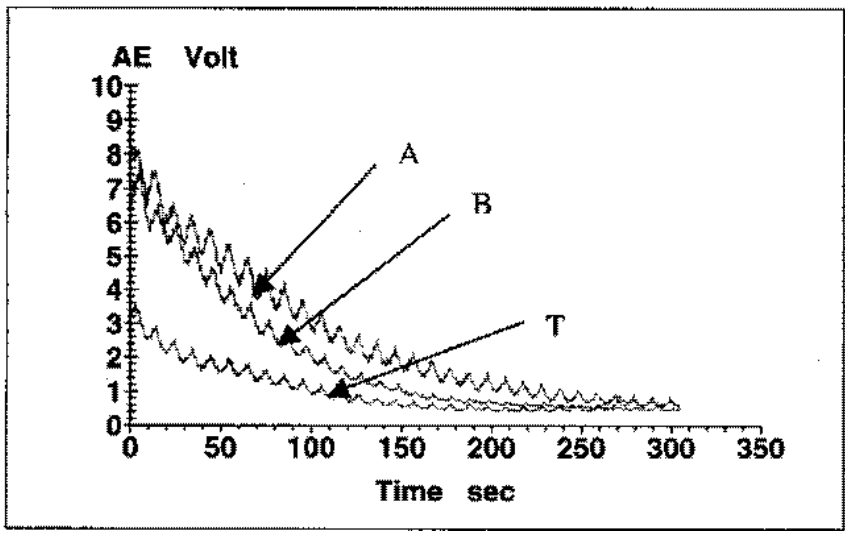


Fig. 4. Variation of AE signal of three different samples while polishing with no upper rotation. Samples were polished with 150 rpm at 3 PSI.

Fig. 5(a) shows the AE signal variation while polishing with active upper rotation (work performed at CETR, Inc. facility) and Fig. 5(b) shows the optical picture of the coupons after polishing. As it is seen for the samples polished with non upper rotational machine, highest AE signal is recorded for sample A and lowest signal for sample TEOS while a little smaller value is recorded for sample B than sample A. As sample A consists of Cu-ultra low-k samples with lowest k values, delamination may be more for this sample. It can be seen from Fig. 5(b) samples get polished more uniformly with an upper rotation whereas samples gets more polished at the leading edge only for no upper rotation. From the samples it could also be seen that all the A and B samples are delaminating whereas TEOS has no sign of delamination. Further analysis of the samples with SEM, shown in Fig. 6, reveals that during the process Cu either gets peeled off or delaminated. Even delamination of underlying interlayer dielectric films could be seen.

In all conditions, and experimental setups AE signal is higher for sample A and B than TEOS. Also, sample A produces more AE signal than sample B. It is prudent to mention that the only difference among the three samples is the underlying ultra low-k ILD film. High AE for A and B sample may be an indication of delamination of Cu from barrier and ILD interface. As Low-k B has slightly higher k values, which implies better mechanical strength [5], sample B shows better resistance to the CMP process. Lower amplitude of AE for sample B than sample A is the reason for this conclusion.

It is found that AE sensor is capable of detecting delamination. We have also noticed that severe delamination or scratching on the surface could be monitored with the AE sensor, which produces spikes during the unusual polishing. Two A samples were run in the same conditions along with a TEOS sample. The expanded AE signals for all three samples are shown in Fig 7(a). Optical and SEM picture of the sample coupons are also shown in Fig. 7 (b) and (c). It can be seen from Fig. 7 (a) that several spikes occur for the sample with visible scratch on it. AE signal for TEOS shows very smooth and reduced noise level. By analyzing the noise level of the AE signal, one can determine in situ characteristics of the polish **[6].**

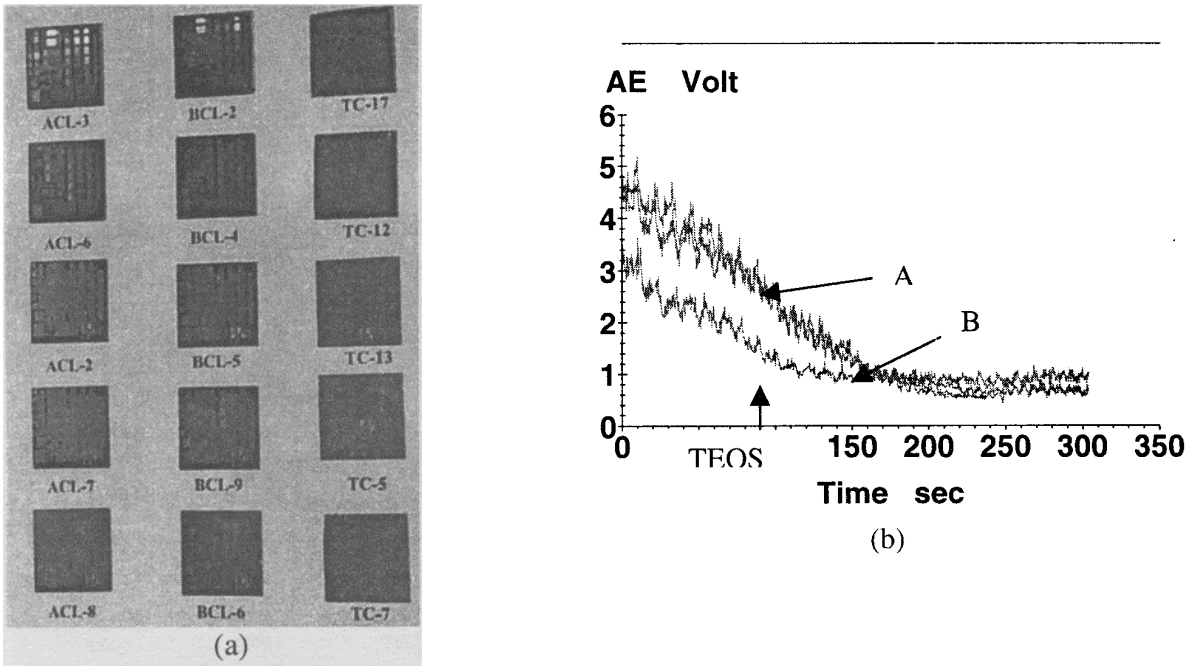


Fig. 5. (a) Picture of sample coupons after polishing. ACL -Pattern low-k A, BCL - Pattern low-k B and TC - Pattern TEOS. Samples were polished with the machine with active upper rotation and (b) Variation of AE signal of three different samples while polishing with active upper rotation. Polishing were performed at contact pressure of 7 PSI, with pad speed 120 rpm, and wafer speed 110 rpm.

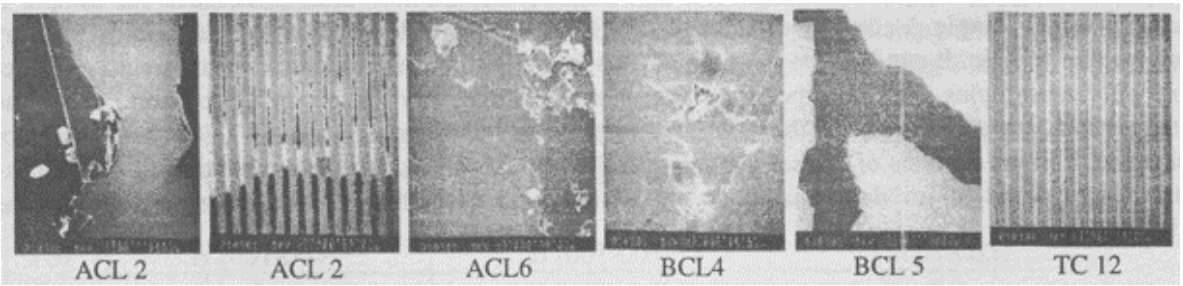
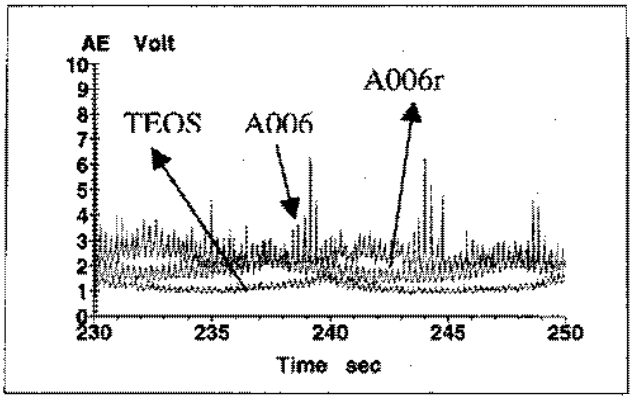
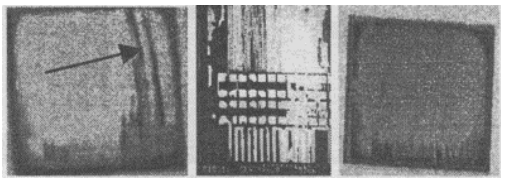


Fig. 6 SEM micrographs showing the different kind of delamination for sample A and B and no delamination for TEOS sample. All the samples were polished on the machine with active upper rotation.



(a)



Optical (A006) SEM (A006) Optical (A006r)
(b) (c)

Fig. 7. (a) Variation of AE signal during polishing in the time interval of 230 - 250 s for three different samples. Peaks are seen for sample A006, (b) picture of sample A006 and (c) picture of sample A006r

Conclusions:

The following conclusions can be made from this study:

- Different patterned samples were tested with active and passive upper rotation
- COF decreases with the increase of both RPM and PSI.
- COF is more for sample A and B than TEOS
- In all cases amplitude of AE signal higher for low-k A than Low-k B and Low-k B than TEOS (A>B>TEOS)
- Higher amplitude is a definite indication of delamination
- Low-k B is slightly better than low-k A
- With upper rotation delamination occurs on the entire surface
- With upper static condition delamination occur at the leading edges only
- AE signal and COF values could be used as an in situ monitoring method for polishing Cu-ultra low-k system
- Bench top polisher is capable of doing initial screening of ultra low-k materials and it could be an ideal R & D tool for CMP process with reduced cost of ownership.

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