

Effect of Propellant Combustion on Sapphire

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ABSTRACT

Sapphire (Al_2O_3) is the window material of choice for laser beam transmission into the combustion chamber of laser-ignited guns. To evaluate the long-term effects of propellant combustion on an Al_2O_3 laser window, it is important to know the window temperature during firing. This paper presents temperature data on an Al_2O_3 sample located in the breech face of the gun where the laser window would be in a laser-ignited 155 mm (M199) cannon. Al_2O_3 sample is a substrate material of a commercially sold thin-film thermocouple, and is therefore thermally, if not optically, representative of an actual Al_2O_3 laser window.

1 INTRODUCTION

Conventional large calibre guns use convective heat transfer from the igniter (tube or pad) to initiate combustion of the main propellant charge. On the other hand, laser ignition, a relatively new technology, uses radiation heat transfer to light the main charge directly.

In laser-based ignition, radiation is transmitted from an outside laser source (typically a *Nd:YAG* or *Nd:glass* laser, which operates between 1.06 μm and 1.05 μm , respectively into the combustion chamber by a fibre-optic cable connected to a laser window located in the breech face of the gun¹. The laser window must, obviously, be transparent to laser radiation, but it must also be capable of withstanding the high pressures and temperatures generated by the main charge combustion. The material of choice for this application is sapphire (Al_2O_3) having a melting point of 2,040 °C and is reputed to be chemically stable at high

temperatures². It also has high strength: At room temperature, it has a tensile strength of ~425 MPa along the optical axis and a compressive strength of ~1,950 MPa. However, at elevated temperature, its compressive strength dramatically decreases³, incurring a 97-98 per cent loss (to ~50 MPa) above 600 °C.

Furthermore, a laser ignition window is subjected to high rates of pressure and temperature change. Pressure changes of 10 MPa/ms and temperature changes of 100 °C/ms are typical. These rates are several orders of magnitude higher than those reported elsewhere in the literature on pressure and temperature rate-induced changes⁴⁻⁶ in the properties of Al_2O_3 .

Although Al_2O_3 -based laser ignition windows have been successfully used at the research level, the long-term effects of propellant combustion on such windows are not well known. Some have noted both cracking and changes in the surface chemistry

on post-fired Al_2O_3 windows*. The objective of the present study is to measure the surface temperature of an Al_2O_3 sample located where the laser window would be in a 155 mm M199 cannon firing various charge configurations. Damage to an Al_2O_3 sample is also reported, and its cause speculated upon, but it is not possible to infer that the damage observed in this sample is indicative of combustion-induced damage that could occur in an actual Al_2O_3 laser window. The reason being, the Al_2O_3 sample used in this study is the substrate of a commercially sold thin-film thermocouple (TC). As such, this sample is of unknown optical character wrt its purity (inclusions, bubbles and defects), surface finish and optical axis orientation, all factors that are reported to affect the strength³⁻⁶ of an Al_2O_3 .

Even though the optical character of the Al_2O_3 -based TC is unknown, its thermal character is identical to that of an actual Al_2O_3 laser window. Thus, this study is unique; it provides the first known source of surface temperatures that can be expected to occur on an Al_2O_3 window due (solely) to propellant combustion.

2. EXPERIMENTAL PROCEDURE

2.1 Instrumentation

A current laser window consists of an Al_2O_3 crystal, 14 mm in diameter, held within a 25 mm hexagonal stainless steel casing (Fig. 1) that threads into the breech face of the gun, as presented in Fig. 2. The window faces the propellant charge when the breech is closed. Alongside the laser window in Fig. 1 is another window-like stainless steel housing that contains a surface-flush K-type TC. This relatively conventional K-type probe was used along with a more exotic, thin-film-on-sapphire TC to measure temperature in the laser window environment.

The thin-film TC was made by vapour depositing a thin-film (0.5-1.0 μm) of rhodium and platinum on an Al_2O_3 crystal, 4 mm in diameter, as

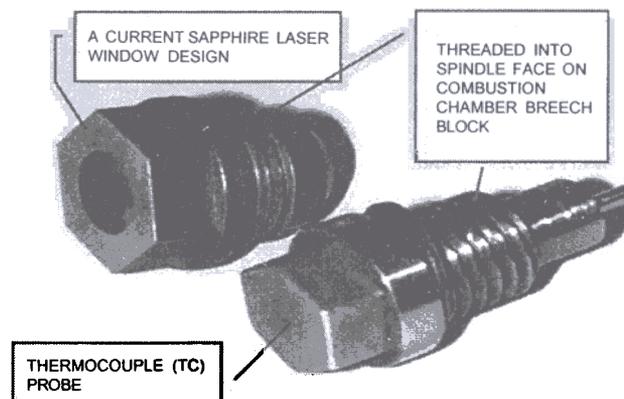


Figure 1. Laser window and one (of two) TC design(s) used in place of window to measure temperature.

shown in Fig. 3**. TC junction was formed in a central region of a thin-film overlap. Wires of platinum and rhodium transmit the temperature-dependent voltage from the bimetallic junction to a signal processing data recorder. Typical of thin-film TCs, the response time is of the order of microseconds⁷. This Al_2O_3 TC provided identical laser window material for duplicating the heat conduction and diffusion effects. Thus, the temperature measurements from this probe were expected to give an accurate assessment of the laser window surface temperature in the gun chamber environment.

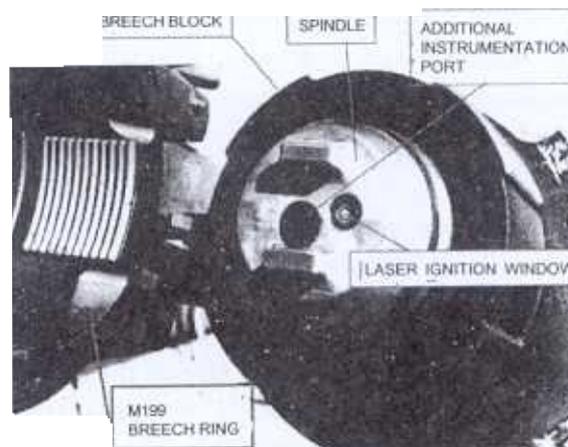


Figure 2. View of the 155 mm (M199) breech ring, breech block, spindle and experimental laser ignition window.

* Kerwien, S.C. Materials analysis and characterisation of a laser ignition system window. US Army Armament Development and Engineering Centre, Picatinny Arsenal, NJ, USA, 1996 (internal report).

** Both types of TC probes were custom made for this test by Medtherm Corp. (Huntsville, AL), a company specialising in the manufacturing of such gauges for the in-bore environment.

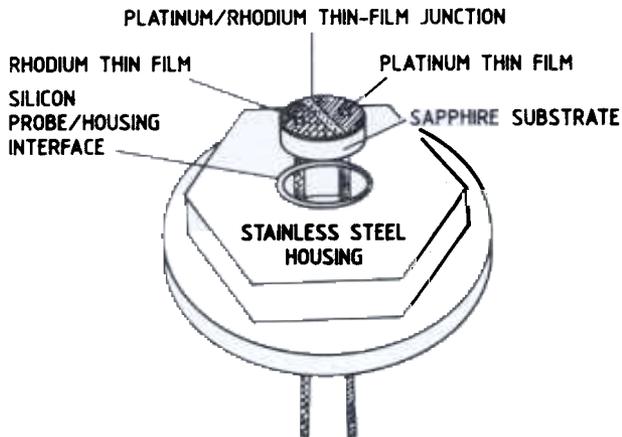


Figure 3. Thin-film-on-sapphire TC probe construction

Figure 4 shows a close view of the spindle component of the breech block, modified to accommodate the 2-TC probes. A broader perspective of the test setup is shown in Fig. 5, where the spindle, breech block, and breech ring are included in the view. TC lead wires were extended through and out the back of the spindle connecting to a data recorder located inside the firing bunker.

In addition to TCs, two copper crusher gauges (M11-type) were used to measure the peak internal combustion pressure in the chamber region for each round fired. Also, the muzzle velocity of each round was recorded with a Doppler radar system (Weibel model-680). These two sources of information (chamber pressure and muzzle velocity) provided an independent check that the firing was typical for a given propellant charge configuration. The peak chamber pressure was also used to assess the potential for crystal damage due to high temperature compression.

2.2 Test Matrix

Temperature data were obtained from a 10 round firing programme, as described in Table 1. In all, there were five different propellant charge configurations (groups); 2 rounds of each type were fired. The five groups spanned a range of propellant combustion conditions from low to high charge. The group I (M3A1 zone 5) is made up of five (unequal) increments (zones 1-5) of bagged M1 granular propellant (flame temperature = 2,176 °C),

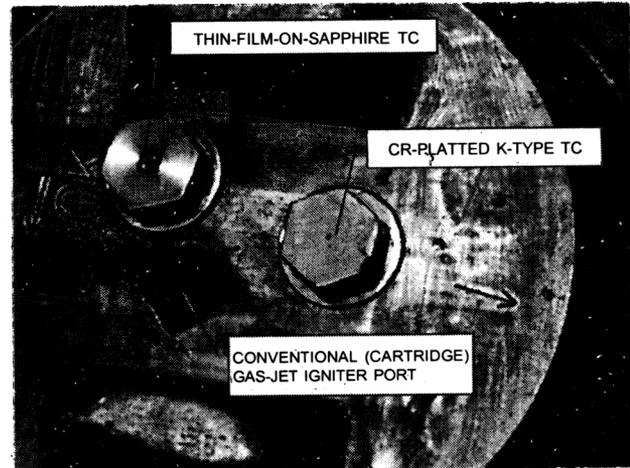


Figure 4. M199 spindle, modified to accommodate 2-TC probes

totalling £ 5.5 (24.5 N). The group II (M4A2 zone 5), consists of three of a possible five bags (zones 3-7) of M1 granular propellant, totalling £ 7.1 (31.4 N). The group III (M4A2 zone 7) uses all the five bags of M1 propellant, totalling £ 13.3 (59.1 N). The group IV, (M119A2 zone 7) is a single bag of M6 granular propellant (flame temperature = 2,298 °C), weighing £ 20.7 (92.1 N). The group V, (M203A1 zone 8) was a single bag of M31A1E1 stick propellant (flame temperature = 2,301 °C) weighing £ 26.3 (117 N). The M101 projectile [weighing £ 96 (427 N)] was used for all firings. The M101 is the (obsolete) predecessor of the M107 projectile, having the same weight, the only difference being in the width of the rotating band.

Table 1. Firing test sequence

Round number (group number)	Propellant charge designation (weight, N)
1(1)	M3A1 zone 5 (24.5)
2(2)	M4A2 zone 5 (31.4)
3(3)	M4A2 zone 7 (59.1)
4(4)	M119A2 zone 7 (92.1)
5(5)	M203A1 zone 8 (117.0)
6(5)	M203A1 zone 8 (117.0)
7(4)	M119A2 zone 7 (92.1)
8(3)	M4A2 zone 7 (59.1)
9(2)	M4A2 zone 5 (31.4)
10(1)	M3A1 zone 5 (24.5)

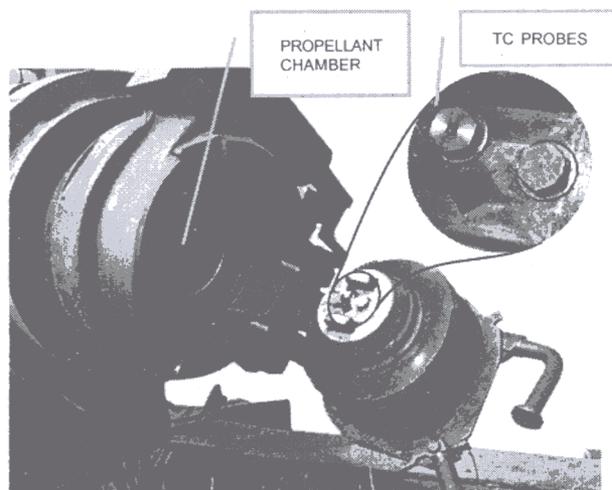


Figure 5. Open breech view of TC probe locations

3. TEST RESULTS

3.1 Chamber Pressure & Muzzle Velocity

The average pressure and muzzle velocity results are presented in Table 2. As is typical for this gun system, the peak chamber pressure (averaged over both chamber gauges) was repeatable to within < 10 per cent for rounds in the same group. It is also typical for the muzzle velocities to repeat within ~1 per cent, as they did. An instrumentation failure prevented a muzzle velocity measurement for round 7. In general, the chamber pressure and muzzle velocity went up with increasing propellant charge weight (Table 1) in going from groups I-V. The only exception to this correlation was group II, which had a lower chamber pressure than group I, even though it had more propellant and a higher muzzle velocity than group I. The explanation lies in the fact that the propellant grain geometry was slightly different for the M1 propellant in group II than group I, giving it a different burn rate history.

3.2 Breech Face Temperatures

In general, rounds were fired every 30 min. Probe temperatures were monitored for several seconds prior to and after firing each round. During the combustion event, temperature data were recorded at a 10 kHz rate. For each firing, there are

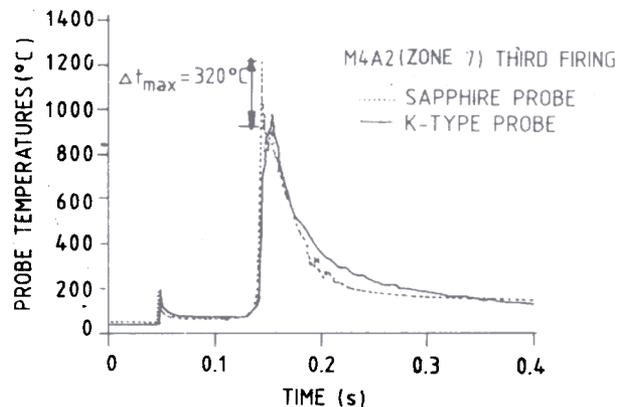


Figure 6. Breech face surface temperatures of both probes for round 3.

2-TC probes (Figs 4 and 5). One probe is a K-type and the other is a thin-film-on-sapphire type. For the first four rounds, the only round where an Al_2O_3 gauge functioned properly was for round 3, with the results shown in Fig. 6. The peak Al_2O_3 temperature of 1,235 °C was 320 °C higher than the K-type probe. Although the peak temperature values may be different in magnitude as shown in Fig. 6, both gauges registered nearly the same initial (primer-igniter) temperature spike as well as virtually the same time to reach the peak temperature, (~100 ms), after ignition.

In literature, there have been measurements reporting that the compressive strength of Al_2O_3 along the optical axis drops to ~ 50 MPa above 600 °C. Table 2 shows that, in all cases, the peak

Table 2. Maximum chamber pressure and muzzle velocity results

Round number (group number)	Chamber pressure (MPa)	Muzzle velocity (m/s)
1(1)	112	369
2(2)	78	396
3(3)	195	569
4(4)	223	696
5(5)	351	848
6(5)	350	846
7(4)	223	No data
8(3)	181	570
9(2)	79	402
10(1)	105	371

chamber pressure was ≥ 75 MPa, and Fig. 6 implies that the peak surface temperature can be well above 600 °C. If the optical axis of the Al_2O_3 TC was, by chance, closely aligned with its surface normal, then the combination of high pressure and temperature would implicate that the Al_2O_3 TC might fail mechanically in this environment. In fact, since only two of the four Al_2O_3 probes procured for this test survived even one round, this may have been the case. Comparatively, the K-type TCs worked well; one K-type gauge operated for nine consecutive rounds.

After the third Al_2O_3 probe failed, with the firing of only round 4 (of ten) rounds, a K-type TC was threaded into the Al_2O_3 port. Thus, for round 5, both temperature sensing probes were of the K-type. Figure 7 shows the temperature recorded on the breech face by the side-by-side K-type TCs. It can be seen that the primer-igniter pulse and the peak temperatures are nearly aligned in time and that the peak temperatures differ by only ~ 55 °C. This 7 per cent difference between adjacent probes of the same type supports the contention that the 30 per cent difference between the K-type and Al_2O_3 TCs in round 3 (Fig. 6) was due primarily to the difference in their thermal properties.

The K-type TC used in the Al_2O_3 TC port for round 5 failed on round 6. In its place, the last Al_2O_3 probe was installed. Fortunately, this probe functioned properly for round 7 (though it failed on

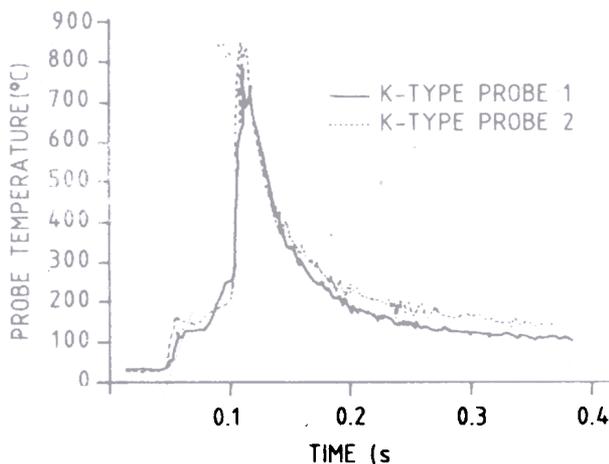


Figure 7. Breech face surface temperature at two adjacent K-type probes, for round 5.

Table 3. Probe-temperature summary

Round number (group number)	K-type TC (°C)	Al_2O_3 TC (°C)
1(1)	755	TC failure
2(2)	785	TC failure
3(3)	915	1235
4(4)	855	TC failure
5(5)	795	850 (K-type TC)
6(5)	955	TC failure
7(4)	1020	1405
8(3)	1030	TC failure
9(2)	1095	TC failure
10(1)	TC failure	TC failure

round 8). Its peak surface temperature was 1,405 °C compared with a reading of 1,020 °C from its adjacent K-type probe (Table 3). The same K-type probe that recorded round 1, recorded all rounds up through 9, but failed on round 10. Table 3 gives a probe-temperature summary for all 10 rounds.

It is apparent from the scatter in peak temperatures (Table 3) that round-to-round variation within the same group is relatively large. For instance, a 55 °C probe-to-probe difference was noted in round 5, whereas a 310 °C round-to-round difference (measured with the same probe) was noted between rounds 2 and 9 (i.e., between the first and second firing of the M4A2 zone 5 charge). On

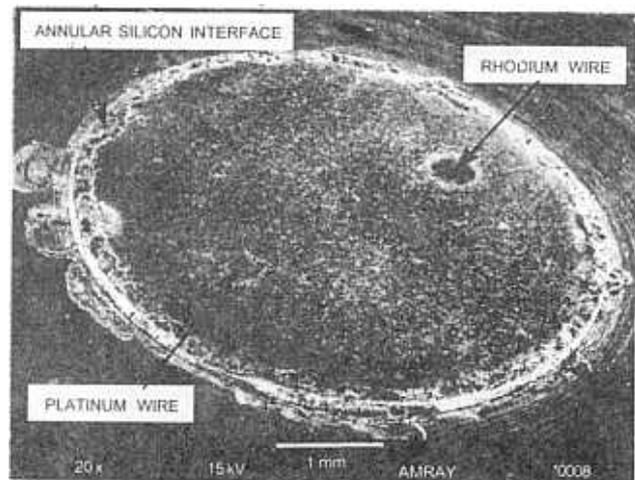


Figure 8. SEM of Al_2O_3 probe surface before firing

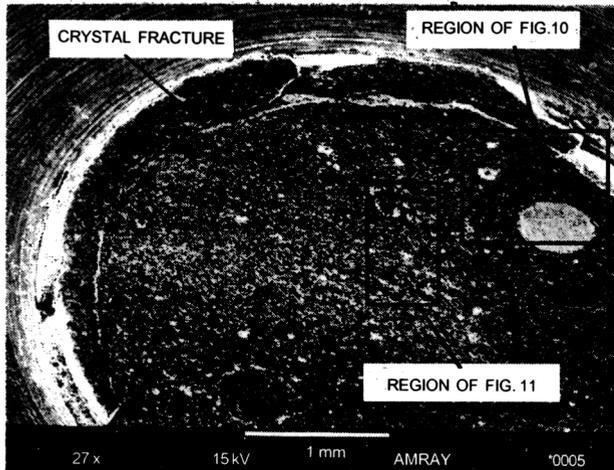


Figure 9. SEM of Al_2O_3 probe surface after firing round 1

an average, the peak temperature for the round 2 in each group was about 185 °C higher than the round 1. There was no significant difference in the initial temperature between round 1 and round 2, nor was there any indication of an increasing temperature bias in the gauge (as concluded from Fig. 7). Hence, at the current time, it is not known why, other than by chance, the round 2 in each group had a higher temperature than the round 1.

3.3 Sapphire (Probe) Surface Damage

Having implied that one possible cause of failure in the Al_2O_3 TCs was mechanical damage to the crystal from the combination of high

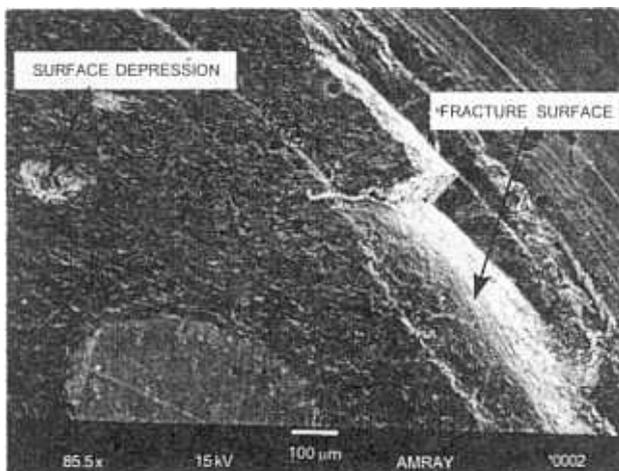


Figure 10. SEM of crystal fracture and crater-like surface depression (magnified from Fig. 9).

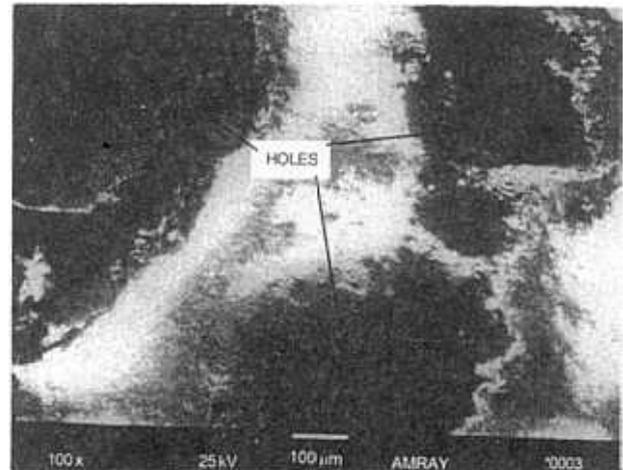


Figure 11. SEM of holes in the post-fired Al_2O_3 surface (magnified from Fig. 9).

temperature and pressure, evidence for this claim is shown in Figs 8-10. Figure 8 is a scanning electron micrograph (SEM), magnified 20x, of the surface of one of the Al_2O_3 TC probes (in Fig. 3), as it looked before firing. Although the rhodium and platinum thin-film coatings are not distinguishable in this image, the rhodium and platinum wires running through the Al_2O_3 crystal are perceptible.

Figure 9 is an SEM (27x) of an Al_2O_3 probe after the firing. Clearly, there is physical damage. For instance, the semicircular cleavage line near the outer edge of the probe face is an evidence of crystal fracture. Figure 10 provides a close SEM view (85x) of the fracture site. Also shown in Fig. 9, but more clearly in Fig. 10, is a small crater-like pit, or surface depression in the crystal, roughly 100 μ m in diameter.

In addition to fracture and pitting, there are hole-like regions on the crystal surface of Fig. 9, magnified in Fig. 11, where it appears that a plug or core of crystal material is missing. These plugs/cores are 20-100 μ m in diameter (depth unknown).

It is conceivable that pits and plugs could be caused by the same phenomenon. Reasoning, after peak pressure, the compressed crystal will spring back to its original shape. However, bonding in the

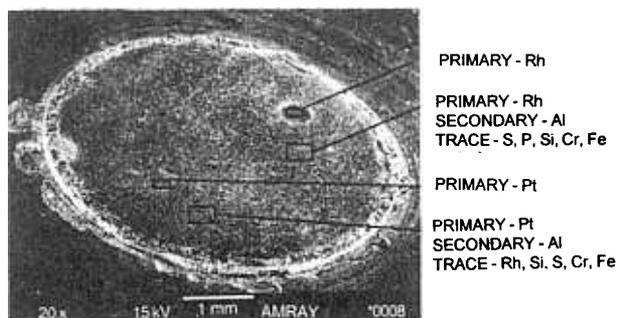


Figure 12. Elemental energy dispersive X-ray analysis of Al_2O_3 probe before firing.

crystal is weakened³ by high temperature[#]. Conceivably, the inertia of the returning surface may exceed the restraining capacity of the bonds in some regions. If so, these regions could break away from the surface, leaving voids that appear as pits and holes.

3.4 Elemental Analysis of Sapphire Surface

Using an energy dispersive X-ray technique, elements above magnesium were identified on the surface of the Al_2O_3 probe. Before firing, the surface constituents were: Aluminium, rhodium and platinum, with trace amounts of sulphur, phosphorus, silicon, chromium, and iron. As expected, the primary element in each half (Fig. 12) was either rhodium or platinum with the secondary element being the substrate material—aluminium. It is not known why trace amount of elements of sulphur, phosphorus, silicon, chromium and iron were found on the surface.

After firing, the percentage of surface area that was primarily rhodium or platinum decreased dramatically, as shown in Fig. 13. Out of five areas sampled sequentially across the platinum-side of the probe, two were found to be primarily aluminium, rather than platinum. Apparently, a significant fraction of the thin-film coatings of platinum and rhodium was removed by the combustion event. This too (along with mechanical damage) is a possible cause of TC failure. A trace amount of potassium was also found on the post-fired surface. This is not unusual, since

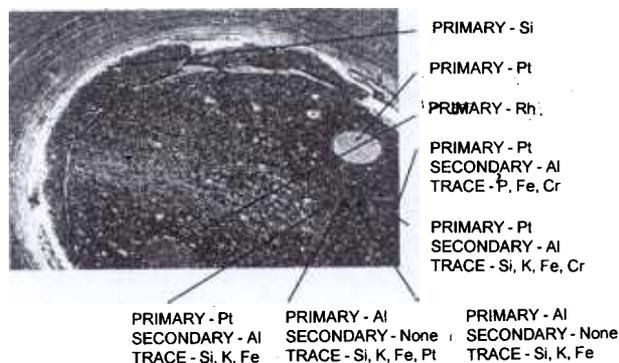


Figure 13. Elemental energy dispersive X-ray analysis of Al_2O_3 probe after firing.

potassium (as well as sulphur) is an element of the propellant.

4. CONCLUSION

Al_2O_3 is the window material of choice for laser pulse transmission (through the breech and into the chamber) in laser-ignited guns. However, very little is known about the thermal response of Al_2O_3 in this environment. This report shows the test results from an experiment, where Al_2O_3 was the substrate of a thin-film TC mounted in the same location as a laser window in a 155 mm (M199) cannon. Various charge configurations were fired (all had propellant flame temperatures above 2000 °C) and the surface temperature measured. The results indicate that an Al_2O_3 laser window will incur a peak temperature exceeding 1000 °C in this environment, regardless of charge configuration.

Physically, the cylindrical-shaped Al_2O_3 probe sustained significant damage with each round fired. Semicircular cleaving of the crystal was observed. In addition, there was surface pitting and plugging. Although, it is not known where the optical axis of Al_2O_3 probe was in relation to the compression axis, the observed damage would be consistent with a near alignment between the two. Since, in that case, the reported reduction in tensile strength at elevated temperature, coupled with decompression (after the peak chamber pressure is reached) could be the cause of both pitting and plugging on the surface.

Harris and Schmidt (1995) not only showed that the compressive strength, but also the tensile strength of sapphire decreased with increased temperature on the optical axis.

In spite of significant damage incurred by these Al_2O_3 samples—after just one firing— Al_2O_3 windows have, in general, proven themselves fairly resilient to multiple firings. The explanation for this discrepancy may lie in details of crystal fabrication (such as axis orientation, crystal purity, and surface finish) for laser windows versus TCs and/or in the method of mounting Al_2O_3 into a laser window fixture.

REFERENCES

1. Barrows, A.W.; Force, B.E.; Chang, L.M.; Howard, S.L.; Beyer, R.A. & Newberry, J.E. Laser ignition testing of two-piece tank ammunition for advanced tank cannon systems (ATACS). US Army Research Laboratory, Aberdeen Proving Ground, MD, 1994. Report No. ARL-TR-474.
2. Schmid, F.; Khattak, C.P. & Felt, D.M. Producing large sapphire for optical applications. *Am. Ceram. Soc. Bull.*, 1994, **73** (2).
3. Harris, D.C. & Schmid, F. Mechanical strength of sapphire at elevated temperature. Proceedings of the 6th DOD Electromagnetic Windows Symposium, 1995.
4. Newcomb, S.A. & Tressler, R.E. Slow crack growth in sapphire fibres at 800-1500 °C. *J. Am. Ceram. Soc.*, 1993, **76**, 2505-512.
5. Breval, E.; Srikanth, V. & Subbarao, E.C. Acoustic emission and microcracking in sapphire, sintered Al_2O_3 , Al/Al_2O_3 composite, and aluminium. *J. Am. Ceram. Soc.*, 1995, **78**, 2541-544.
6. Chervin, J.C.; Syfosse, G. & Besson, J.M. Mechanical strength of sapphire windows under pressure. *Rev. Sci. Instrum.*, 1994, **65**, 2719-725.
7. Medtherm Corporation. Measure surface temperature with a response time as little as 1 microsecond. *Bulletin 500*, 1994, Huntsville, AL, USA.

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