MACRO- AND MICROSTRUCTURAL ANALYSIS OF TATB SAMPLES SUBMITTED TO A COMBINED THERMAL / MECHANICAL AGGRESSION

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The effects of initial thermal damage on shock sensitivity are investigated for TATB-based high explosive. Two kinds of thermal aggression have been considered: heating (from 220°C to 250°C during 1h to about 7 hours) and heating then cooling to ambient temperature. Then, three types of plate experiments have been performed in the shock-to-detonation (SDT) pressure range: high level plate impacts to measure the modified initiation depths and material velocities, lower level plate impacts and aquarium tests to recover the damaged samples for microstructural analysis.

The experimental results have been used to assess the influence of thermally pre-damaged samples on TATB SDT macroscopic characteristics. The microstructural investigations have identified damages due on one hand to the thermal aggression only and on the other hand to the combined thermal/mechanical stimulus. The localization and evolution of a series of phenomena from grains deformation to reaction zones have been determined as a function of temperature, heating duration and pressure. These data have then been analyzed to attempt to quantify the effects of these thermal and mechanical aggressions on each identified microstructural phenomenon.

INTRODUCTION

Sensitisation to mechanical impacts of TATB-based high explosives by preheating has been investigated for many years in the field of safety studies in particular in the case of a fire. Characterization of this sensitisation was previously performed [1] for US TATB-based explosives mainly in terms of initiation depth.

This present work has been realised in support of CEA for its expertise of situations when explosives or munitions are first heated up and cooled down. It concerns the characterization of a French TATB-based explosive developed in CEA Laboratory. The CEG work has been split in two experimental phases, the first one being devoted to compare the sensitisation results of this heated explosive to those obtained from the US explosive [1], the second one considering the production and the microstructural evolution of TATB samples preliminary heated and cooled down and then mechanically stimulated.

For the first phase, a heat generator has been developed and adapted to a powder gun in order to heat TATB samples up to 250°C before submitting them to plate impacts. The diagnostic is either pop-plot configuration to get the initiation depth of such materials or Doppler Laser Interferometer to get material velocity profiles before detonation.

The second phase has been realised to assess the sensitisation to mechanical stimulus of TATB samples preliminary heated up and then cooled down. The same tests as in the first phase have been performed on this type of material and the results have been compared to these obtained from only heated samples.

The main task in this phase however has consisted in recovering heated/cooled and then mechanically aggressed samples for microstructural analysis. Some of them have been directly observed with an optical microscope to determine the microstructural damage due to the thermal aggression only. The others have been submitted to aquarium tests for a non sustained shock propagation or to plate impact tests for a sustained shock propagation, and then were recovered for microstructural observations to assess the additional damage due to the mechanical stimulus.

With this type of diagnostic, sensitisation to impacts of only preheated or preheated then cooled TATB explosives could be assessed:

- macroscopically by experimental determination of initiation depth and material velocity profiles as a function of the thermal aggression type or impact pressure.
- microscopically by the analysis of the microstructural evolution as a function of the temperature, the thermal plateau duration, the pressure level and the pressure profile.

SAMPLES TREATMENT

TATB samples are thermally then mechanically stimulated, with different methods depending upon the type of investigation chosen for each sample: macro- or microstructural characterizations.

All the used methods either for the thermal aggression or for the mechanical aggression are presented in this paragraph.

Thermal cycling methods:

T1 Type: Heated then cooled samples:

This method is used to recover samples for microstructural investigations.

In a specific oven the samples of a diameter of 30 mm and a thickness of 8 mm are submitted to a temperature ramp up Tmax (220°C≤Tmax≤250°C) at a velocity of 5°C/mn then to a plateau at Tmax with a duration of t_p ($1h \leq t_p \leq 7h$). The samples are then cooled by cutting off the oven (Figure 1).

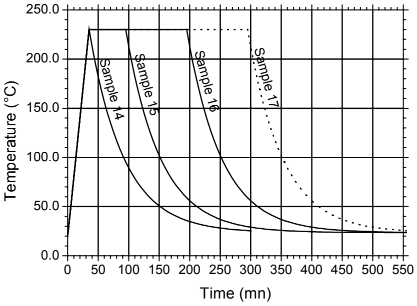

Figure 1: Examples of heating/cooling treatments (Tmax=230°C)

T2 Type: Only heated samples:

This method is employed for heating samples of a diameter of 75 mm and a thickness of 30 mm just before a plate impact.

The heating device has been adapted to the CEG Ares powder gun which will be used for plane impact experiments and is mainly constituted of a hot air generator (up to 280°C at a heating rate from 0.01°C/mn to 15°C/mn) and an insulated pipe guiding the heated air towards the gun muzzle (Figure 2).

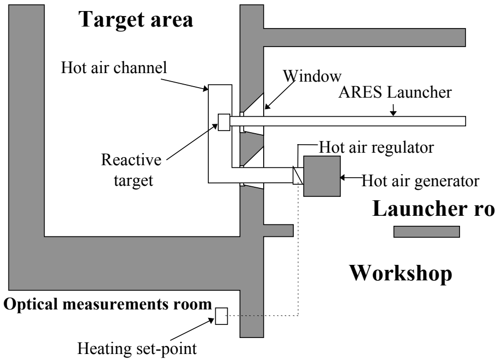

Figure 2: Target heating device adapted to CEG Ares launcher

The same types of heat treatment as previously described may be applied to the TATB samples with this device.

Mechanical aggression methods:

These methods aim to submit a shock level between 4 and 100 kbars to TATB samples either with their recovering (heated then cooled samples) or with no recovering (only heated and heated then cooled samples).

M1 Type: Sustained shock with no recovering device:

CEG Ares powder gun is used to launch a plane teflon impactor against the TATB-based target. The experimental set-up is described in Figure 3.

The impact pressure is given from the impactor velocity and is in the range [40-100 kbars].

The main instrumentation associated to this configuration is a Doppler Laser Interferometer through the measurement window to get material velocities as a function of the impact pressure or a pins array on a sample wedge to determine the run distance to detonation also as a function of the impact pressure. In this case, the explosive target is not a simple cylinder but a truncated one with a 30° wedge along which the pins are aligned (Figure 4).

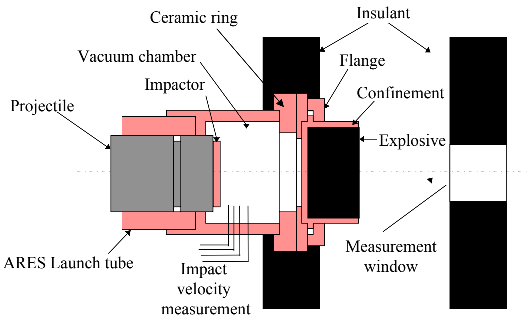

Figure 3: *Experimental set-up for sustained shock with no sample recovering system*

Figure 4: *TATB-based target instrumented by pins array*

M2 Type: Sustained shock with recovering device:

As previously plate impacts technique is applied to generate a sustained shock in the TATB-based targets, but, for this method, a gas gun (CEG Deimos launcher) is used to which a samples recovering device has been adapted.

The target and impact configurations are given in Figure 5.

TATB high explosive (diameter 32 mm, width 5 mm) is confined in this sample holder, enclosed by a teflon cup. The release waves are trapped in lateral and back teflon plates.

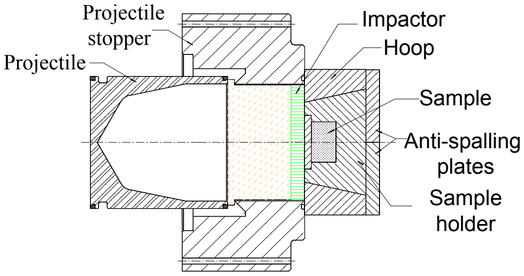

Figure 5: *Experimental set-up for sustained shock with sample recovering system.*

The shock conditions induced by the sustained plane impact tests are between 6 and 11 kbars in the high explosive targets. The lateral release waves reach the TATB material about 10 μs after the impact.

A recovery device (Figure 6) allows to get the shocked TATB sample holder without introducing over-damages after the shock. The system is constituted of a series of materials with increasing densities.

Figure 6: *Samples recovering system associated to CEG Deimos gas launcher*

M3 Type: Unsustained shock with recovering device:

A non-sustained shock may be generated in TATB samples with the aquarium test technique. This technique consists in creating in a water tank, by detonation of a spherical octoviton booster, a shock wave which will propagate through immersed samples (Figure 7).

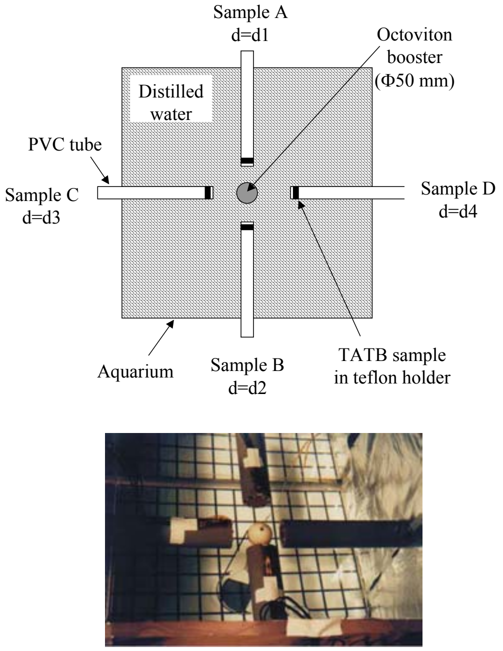

Figure 7: Schemas and photograph of the experimental set-up for generating non-sustained shock with samples recovering

The pressure levels inside the samples are given from the distance d between the samples and the booster and measured with pressure gauges . In this study, they are in the range [4-50 kbars].

Once the shock wave has propagated in the samples they are ejected through out the PVC tubes and recovered outside the aquarium in polyurethane blocks.

Microstructural analysis:

This task has been performed through a contract with "Sciences et Applications" Society, (Le Lafayette - 85, Avenue Kennedy - 33700 Bordeaux Merignac - France) and carried out by P. Lambert.

Recovered samples from T1-type thermal treatment and from combined T1-type thermal treatment and M2- or M3 - mechanical stimulus methods have been submitted to microstructural analysis to identify damage evolution as a function of temperature and/or shock levels. This analysis has been based on microstructural observations of TATB microsections using a reflection optical microscopy technique. This method has already been applied at CEG to assess the microstructural evolution of pressed TATB submitted to different kinds of insults (cook-off tests, "hot needle" test, frozen combustion, ultrasonic waves, drop-weight impact) [2].

RESULTS

NON-RECOVERED SAMPLES

Pop-plot

Two TATB samples have been submitted to M1 type mechanical stimulus once they have been heated, at 0.75°C/mn, up to 230 °C during 1 or 2 hours.

For comparison, the same type of plate impact has been applied to a sample at ambient temperature and to another sample which had been pre-heated at 230°C during 7 hours and then cooled down to ambient temperature (T1 type thermal aggression).

The associated instrumentation is a pins array to determine the run to detonation time and distance.

Results are given in Table 1.

The experimental run to detonation distance values and other US experimental values related to PBX9502 compositions have been implemented [1] in the same graph for comparison (Figure 8).

Table 1: Pop-plot results

Shot n°	**CA00073**	**CA01009**	**CA01012**	**CA01018**
Thermal treatment	1h @ 230°C	2h @ 230°C	Ambient Temp. (12°C)	Heated at 230°C during 7h then cooled (*)
Shock level (kbar)	64.7	48.6	130.1	100.4
Shock velocity (m/s)	4059	3580	5379	4797
Deto. velocity (m/s)	7557	7679	7687	7083
Run to deto. Time (µs)	1.67	3.67	1.77	2.24
Run to deto. Distance (mm)	6.79	13.14	9.55	10.74

(*) The shot has then been performed at 4°C.

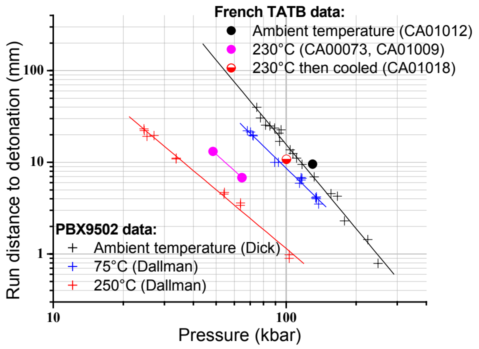

Figure 8: Experimental run to detonation depths compared to PBX9502 results.

Penetration depths of ambient temperature or heated French TATB samples are consistent with PBX9502 results. Sensitisation by heating increases with temperature.
Sensitisation of a sample preheated during 7h at 250°C then cooled appears similar to a sensitisation by heating at about 75°C.

Material velocities

The aim of this trial was to approach another way to illustrate the influence of the thermal aggression level on the TATB shock to detonation transition characteristics. For that, the mechanical stimulus M1-method has been used, associated to a Doppler-laser interferometer (DLI) diagnostic at the rear of the TATB sample. A samples thickness of 13.14 mm has been chosen from pop-plot results (Figure 8) to ensure that they would not detonate for impact pressures below 48.6 kbars. M1-method has been applied to generate pressure inputs of 30 and 40 kbars on two samples heated at 230°C, and on a sample heated at 230°C then cooled down to ambient temperature.
DLI records are presented in Figure 9.

The heat up to 230°C then cool down to ambient temperature treatment induces a sensitisation of TATB, which is not negligible even if it is much lower than in the case of a heating at 230°C. This observation is consistent with the pop-plot results.

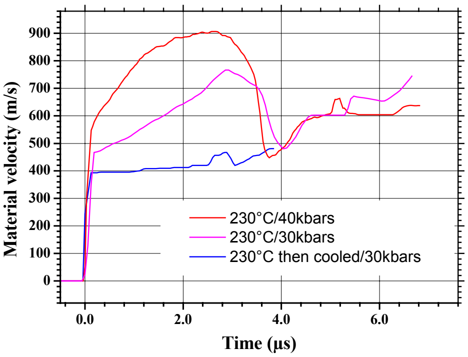

Figure 9: DLI signals for SDT characterization

RECOVERED SAMPLES

Many TATB samples have been recovered after a thermal treatment only (T1-method) or after a combined thermal (T1-method) and mechanical (M2 or M3 type) stimulus to be then submitted to microstructural analysis. The list of the samples, associated to their type(s) of aggression is given in Table 2.

Table 2: Recovered TATB samples for microstructural analysis

TATB sample N°	Thermal aggression	Mechanical aggression		
	Cycle	Method	Pressure (kbar)	Shot number
6	220°C-2h		0	
7	220°C-6h		0	
8	227°C-3h30		0	
9	230°C-3h30		0	
10	230°C-3h30	M3	49.5	ZD00059
12	230°C-3h30	M3	15.6	ZD00059
13	230°C-3h30	M3	8.95	ZD00059
15	230°C-1h		0	
16	250°C-7h		0	
17	230°C-7h		0	
18	230°C-1h	M3	49.5	ZD00064
19	230°C-1h	M3	33.1	ZD00064
20	230°C-1h	M3	15.6	ZD00064
21	230°C-1h	M3	8.95	ZD00064
22	230°C-7h	M3	49.5	ZD00068
23	230°C-7h	M3	33.1	ZD00068
24	230°C-7h	M3	15.6	ZD00068
25	230°C-7h	M3	8.95	ZD00068
29	230°C-7h15		0	
30	250°C-7h15		0	
31	250°C-7h	M3	15.6	ZD01010
32	250°C-7h	M3	8.95	ZD01010
33	250°C-7h	M3	7	ZD01010
34	250°C-7h	M3	4	ZD01010
35	250°C-7h	M2	7.8	CD01007

The microstructural observations of these samples lead to the identification of the microstructural damage as a function of temperature, heating duration, and for the combined aggression, pressure level.

As temperature increases from 220°C to 250°C:

- Disappearing of the initial intragranular porosity, and, locally, appearing of "recristallisation" domains in the grains (Figure 10).
- Appearing of pores at grains boundaries (Figure 11)
- Appearing of reaction zones between the grains (Figure 12).
- Appearing of reaction zones along cristallographic planes and other intragranular discontinuities (Figure 12).
- Extending of reaction zones in the grains (Figure 13).

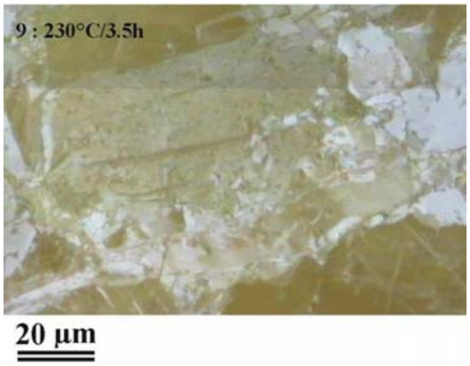

Figure 10: TATB heated at 230°C during 3 h and 30 mn then cooled - Disappearing of intragranular porosity by creation of recristallisation domains (white zones on the figure)

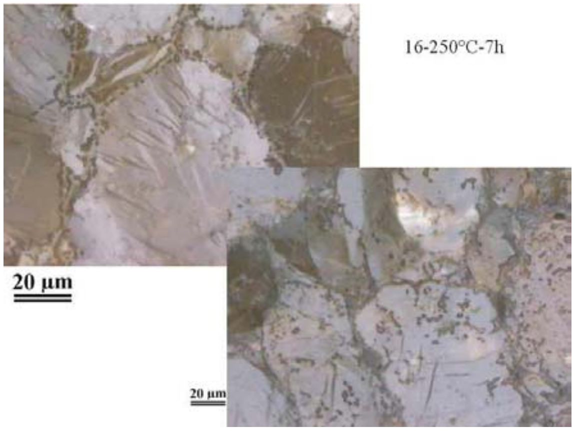

Figure 11: TATB heated at 250°C during 7 h then cooled - Double "dashed lines" of porosity along the grains

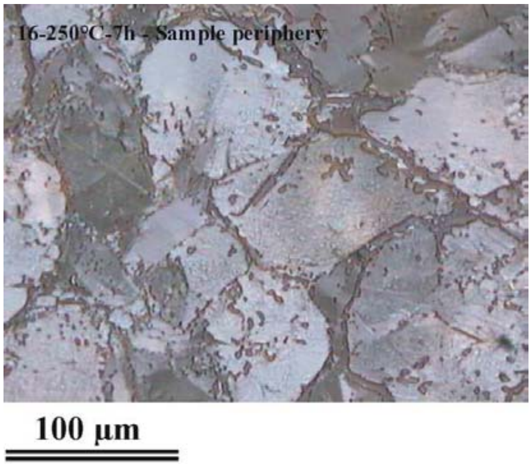

Figure 12: TATB sample heated at 250°C during 7 h then cooled - Coalescence of the double dashed lines of porosity to get reaction zones along the grains; reaction zones also along the recristallisation domains inside the grains.

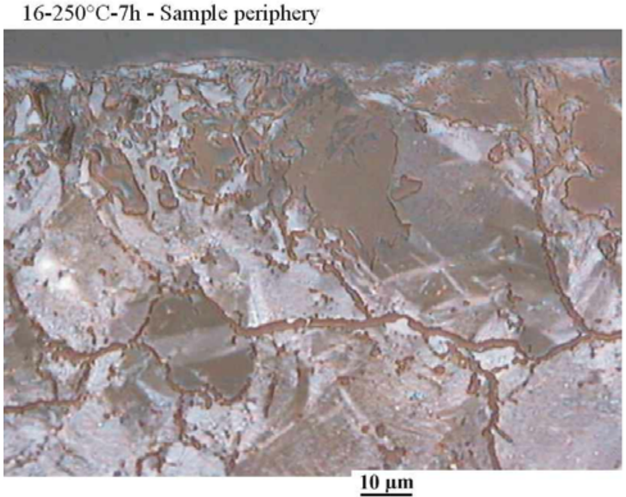

Figure 13: TATB sample heated at 250°C during 7 h then cooled - Grains consumption (reaction zones) from inside and outside the grains.

As pressure increases from 4 to 49.5 kbars:

- Appearing of strain bands in the grains (Figure 14).
- Deformation of the grains themselves (Figure 15)
- Appearing of reaction zones by first initiation at grains boundaries, then at intragranular discontinuities and finally generalization over the grains (Figure 16).

These phenomena are the same for each type of thermal aggression, but the higher the initial thermal aggression is the lower the pressure levels at which reaction mechanisms appear are.

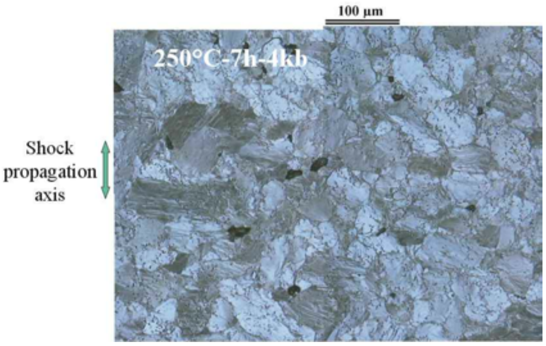

Figure 14: TATB sample heated at 250°C during 7 hours then cooled and impacted at 4 kbars - Appearing of deformation bands in the grains.

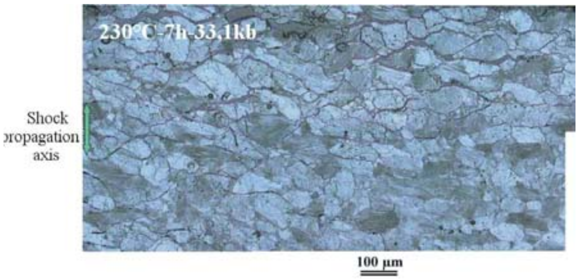

Figure 15: TATB sample heated at 230°C during 7 hours then cooled and impacted at 33 kbars - Flattening of the grains orthogonally to the shock direction axis.

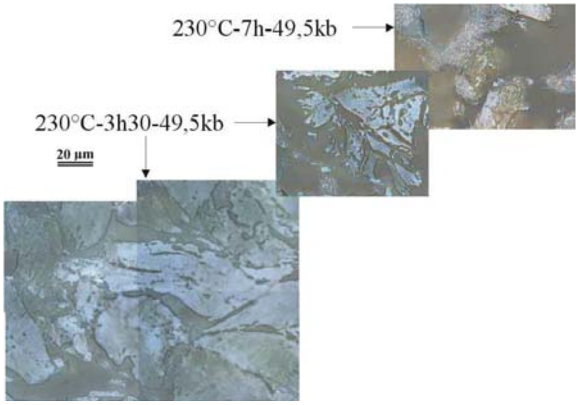

Figure 16: TATB samples submitted to 49.5 kbars impact - reaction zones at grain boundaries and then grains consumption from inside and outside the grains.

All of these observations have come out from first an analysis of each sample and secondly an overall analysis of the trends as a function of temperature, duration of heating and pressure.

A second step of the study was to attempt to quantify all these microstructural evolutions as a function of the same parameters.

The considered microstructural phenomena are porosity, deformation bands density, deformation bands orientation, grains form, grains orientation and specific surfaces.

This work is in progress, and only one example of result is here given concerning the deformation bands orientation. For each microstructural photography, the deformation bands have been counted and sorted in angular sectors with regard to the shock propagation axis.
This method has lead to focus on the effect of the type of mechanical aggression (Figure 17) and on the effect of the heating duration and pressure levels (Figure 18) on the deformation bands orientation.

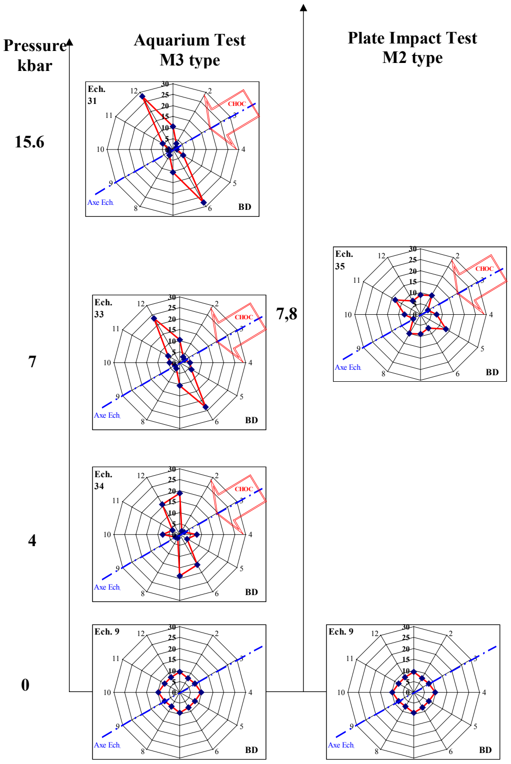

Figure 17: Effect of the type of mechanical aggression on the deformation bands orientation for a TATB sample initially heated at 250°C during 7 hours then cooled down to ambient temperature.

Figure 17 indicates that pressure levels produced from test aquarium tests induce a more important effect on deformation bands orientation than the same pressure levels produced by plate impacts.

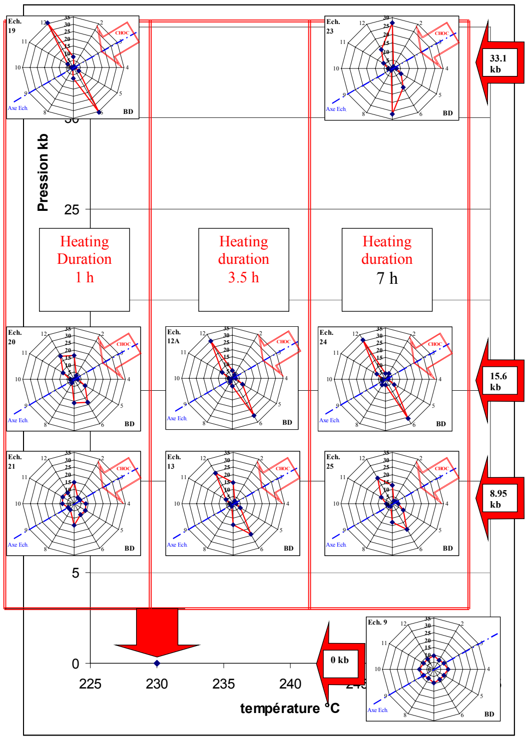

Figure 18: Effects of heating duration and pressure levels (induced by aquarium tests) on the deformation bands orientation for a TATB sample initially heated at 230°C then cooled down to ambient temperature.

Figure 18 confirms the obvious influence of increasing pressures on deformation bands orientation. It also shows that a heating duration between 1 and 3.5 hours emphasizes this phenomenon and has no effect from 3.5 hours to 7 hours.

CONCLUSION

In order to understand shock sensitisation of TATB-based high explosives plate impact and aquarium tests have been performed on initially thermally aggressed TATB samples.

Different experimental methods have been applied to generate a thermal loading (temperatures from 220°C to 250°C, heating duration from 1h to about 7h). Mechanical aggressions have then been conducted on either heated or heated then cooled samples.

Macroscopic data like run to detonation depths and times and material velocities for impact pressures from 40 to 100 kbars have been obtained and compared to US results .

The main part of the study has consisted in recovering TATB samples submitted to a thermal aggression only or a combined thermal/mechanical (between 4 and 50 kbars) loading. A micro- structural analysis of each sample has identified the microstructural damages due to each kind of loading (thermal or combined thermal/mechanical aggression). As temperature increases the grains aspect is modified by vanishing of the porosity and creating of crystallisation domains. The reaction mechanisms are then initiated by a consumption inside the grains first along the boundaries and then along the crystallisation domains.

For each thermal loading, pressure effects are plastic deformation phenomena in and of the grains and the reaction zones are also either at grains boundaries or along the crystallisation domains. These damages appear at a lower pressure when the thermal aggression is higher.

A further analysis aims to assess the evolution of these damages as a function of temperature, heating duration and pressure. This last work is on progress.

All these results will then be provided to CEA in order to validate or improve modelling tasks of TATB decomposition and sensitisation.

REFERENCES

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[2] G. Demol, P. Lambert, H. Trumel, "A Study of the microstructure of pressed TATB and its evolution after several kinds of insults", 11th International Detonation Symposium, Snowmass, 1998.