Low temperature mechanical dissipation measurements of silicon and silicon carbide as candidate material for DUAL detector.

M Bignotto$^1$, M Bonaldi$^2$, M Cerдонio$^1$, L Conti$^1$, G de Paoli$^1$, L Ferrario$^3$, N Liguori$^1$, A Maraner$^4$, E Serra$^3$, L Taffarello$^5$, J P Zendri$^5$

$^1$Department of Physics, Padova University, Via Marzolo, 8, 35100, Padova, Italy and INFN Padova.
$^2$IFN, CNR-ITC and INFN, Gruppo Collegato di Trento, I-38050 Povo, Trento, Italy.
$^3$Fondazione Bruno Kessler, MTLab, Via Sommarive, 18, I-38050, Povo, TN, Italy.
$^4$Laboratori Nazionali di Legnaro (LNL), INFN, Viale dell’Università, 2, 35020, Legnaro, Padova, Italy.
$^5$Istituto Nazionale di Fisica Nucleare (INFN), Sezione di Padova, Via Marzolo, 8, I-35131, Padova, Italy.

E-mail: nicola.liguori@pd.infn.it

Abstract. We present the results of measurements of mechanical dissipations in silicon and silicon carbide samples within the 2-300 K range of temperature. These materials are possible candidates for the sensitive mass of DUAL detector. We have investigated sintered and infiltrated Silicon Carbide (SiC) and P-doped Silicon (Si) in flat plates and cantilevers. Moreover the dissipation of bonded P-doped silicon wafers is scheduled for measurement in the 2-300 K range for the next cryogenic run. We tested a nodal suspension with sapphire and inox steel spheres for flat plates within the same temperature range. We developed two different kinds of capacitive readout: electrostatic comb one for semiconductor silicon and plane capacitor-like for conductor silicon carbide. Moreover an optical lever readout was employed to measure loss angle on SiC cantilevers and silicon disks.

1. Introduction.

The DUAL collaboration is investigating a novel detection scheme, the “dual” detector, which would provide both high sensitivity ($\approx 10^{-23} Hz^{-1/2}$) and wide bandwidth (1-5 kHz) ([1], [2], [3]). The proposed R&D was approved by INFN for a duration of three years and its results will be the demonstration at prototype level of the relevant technologies needed for the realization of the readout performing a complete study of the most critical issues, i.e.

- detector design (limit sensitivity and noise control),
- readout system (geometry and noise to the quantum limit),
- test mass development (choice of the optimal material and fabrication method).

The work presented in the poster enters in the third point. We measured the mechanical losses of some materials which are possible candidates for the manufacture of the DUAL sensitive test
mass. At present the materials under investigation by the DUAL collaboration are sintered α—
and infiltrated C- Silicon Carbide (SiC) and mono-crystalline silicon. We measured the loss
angle of these materials in the range 2-300 K. The measurements were made at the Legnaro
National Laboratories (LNL) cryogenic Test Facility (TF), which is a test site for assembling
and measuring several samples in a single cryogenic (or ultra-cryogenic) run.

2. DUAL sensitivity optimisation.
The material to be chosen has to satisfy few requirements in order to optimise the detector
wide bandwidth and sensitivity. It can be shown that the detector sensitivity scales as the ratio
$Y^2/\rho$, where $Y$ is the Young modulus and $\rho$ the density of the material. This is true under the
following hypothesis:

- the readout of the system is optimised at the Standard Quantum Limit (SQL),
- the topology of the sensitive mass is fixed as cylindrical shape;
- thermal noise contribute is not included in the calculations.

On the other hand, in the case of structural damping, the thermal noise contribution to the total
detector sensitivity is proportional to the product $T\phi$ [4]. Here the choice of the material for the
test mass takes a very crucial role in the sensitivity optimisation. The requirement $T\phi \leq 10^{-8}$
K looks like a good compromise between high sensitivity (of the order of few $10^{-23} Hz^{-1/2}$) and
reasonable feasibility in terms of machinability.

Moreover, fixed the sensitive wide bandwidth (at present around 1-5 kHz), the material
physical properties determine the size of the sensitive test mass of the detector. Commercial
feasibility and/or machinability impose a limit in the available size depending on the material
properties and the factory production capabilities. At present the maximum size available for
the cylindrical topology is about 3m in length and 1m in diameter.

In short the requirements on the candidate materials are

- high value of the ratio $Y^2/\rho$,
- $T\phi \leq 10^{-8} K$

In order to have a more practical idea about the properties of various materials and to compare
them easy each between other, we usually refer to the well known physical properties of Al5056.
In general, if we consider two materials $A$ and $B$ with Young modulus $Y^{A,B}$ and density $\rho^{A,B}$,
and if we optimise the sensitivity curve $S_{hh}$ over the same frequency window for both the
materials $A$ and $B$, then the ratio between the two sensitivities is

$$
\frac{S^A_{hh}}{S^B_{hh}} = \left( \frac{Y^A}{Y^B} \right)^2 \left( \frac{\rho^B}{\rho^A} \right)
$$

In the Table 1 different materials are compared to Al5056. The table collects present knowledge
on dissipation at low temperature. In particular the ratio $S^{mat}_{hh}/S^{Al5056}_{hh}$ is shown. As discussed
above, large size and mass are required to the sensitive mass to achieve wide bandwidth and high
sensitivity in the kHz range. Bonding is needed when commercial available size is not enough
to accomplish requirements. Measurement of bonding contribution in the total dissipation is
crucial to understand if the material can assure the design sensitivity.

3. The LNL facility.
The TF is located inside the same main AURIGA building at LNL, Padova, Italy. The TF
is a completely equipped site for assemblage and measurement of several samples in a single
cryogenic (or ultra-cryogenic) run. In fact, the site is skilled for loss angle measurements and it
Table 1. DUAL detector sensitivity $S_{hh}$ evaluated for different materials and compared with the sensitivity evaluated for Al5056, the material of bar detectors operating. $S_{hh}$ is optimized over the same frequency window for all materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>$\frac{S_{mat}}{S_{hh,Al5056}}$</th>
<th>$\phi$ (low temp)</th>
<th>Constraints</th>
</tr>
</thead>
<tbody>
<tr>
<td>Infiltrated SiC</td>
<td>10</td>
<td>$&gt; 10^{-6}$</td>
<td>Feasible in large dimensions.</td>
</tr>
<tr>
<td>Sintered SiC</td>
<td>27</td>
<td>$&gt; 10^{-6}$</td>
<td>Not Feasible in large dimensions.</td>
</tr>
<tr>
<td>Beryllium</td>
<td>24</td>
<td>$10^{-6}$</td>
<td>Expensive.</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>6.8</td>
<td>$10^{-7}$</td>
<td>Not machinable in large dimensions. Bonding needed.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Available samples at LNL now.</td>
</tr>
<tr>
<td>Mono-Crystalline Silicon</td>
<td>4.4</td>
<td>$&lt; 10^{-8}$</td>
<td>Bonding needed. (Max diam. $\sim$350 mm (USA). Mass limit $\sim$ 300-400 Kg.</td>
</tr>
<tr>
<td>Poli-Crystalline Silicon</td>
<td>(?)</td>
<td>(?)</td>
<td>Available in slightly larger size.</td>
</tr>
</tbody>
</table>

is equipped with a dilution refrigerator and a PID temperature controller so that the available temperature range is $0.02$–$300$ K ($0.02$–$3K$ using the dilution refrigerator + heater, $3$–$70K$ using liquid helium bath + heater, $70$–$300K$ using liquid nitrogen bath + heater). The refrigerator cooling power is about $3$ mW @ $110$ mK. In Figure 1 a section of the whole IVC is shown. The

![Figure 1. A section of the Internal Vacuum Chamber (IVC).](image)

strong points of the TF are its high versatility and short thermalization time. In fact, with its $\approx 0.004m^3$ volume and $50$ kg available payload, it allows to measure several samples in the same cryogenic (or ultra-cryogenic) run. The assembly operations are simplified making as easier as possible mounting several samples in the same run. A pair of days is the time needed to cool down to $4K$ the whole mass (structures + samples).

A chain of cryogenic mechanical suspensions damps the seismic noise allowing sensitive measures up to the thermal noise of a high quality factor (exceeding $10^6$) mechanical oscillator at low temperature. The measured mechanical attenuation of the cryogenic suspensions is $150$ dB @ room temperature. This value is in good agreement with the Finite Element Methods (FEM) analysys and with the constrain to lower the seismic noise below the thermal one (120
dB at 1 kHz, in the case of mechanical transducers, as in the original design). In the TF preliminary runs, overall attenuation was measured applying a driving force on the top floor at the frequency of 1 kHz and reading the output displacement signal of a mechanical transducer at the temperature of 4K. An attenuation of about -180 dB was measured [5].

The core of this apparatus is the Internal Vacuum Chamber (IVC). It houses the cryogenic mechanical suspensions and all the samples to be tested. On IVC top an optical window is placed and an optical bench is implemented so that loss angle measurements are possible also by optical lever besides the usual capacitive readout. The IVC vacuum seal assures a pressure inside in the $10^{-7}$ mbar scale.

4. Measurements and results.

Samples under investigation are both cantilevers and disks for SiC and only disks for Si. Both SiC and Si thin flat plates are mounted employing the nodal suspension. Since for many normal modes the center is a fixed point, nodal clamping should assure high de-coupling between disk modes and clamping motion. Nodal suspension is fulfilled clamping the plate at centre between two spheres housed in a draft which is through or blind central hole. The spheres are in sapphire for silicon and in steel for SiC disks. To avoid to reach the maximum tensile stress during assembling, a soft steel spring is inserted between the beam and the piston (see Figure 2 for some details). This spring assures about 1 N maximum strength.

TF is equipped with four capacitive and one optical readout lines. Capacitive readout is fulfilled making a plane capacitor whose the grounded plate is the sample itself. This works if the sample is an electric conductor like for example SiC. We implemented a so called "multistrip" comb capacitor to realize a displacement readout for dielectrics samples like for example silicon. In Figure 2 a sketch of clamping system and readout is shown (see next section for a brief description), while in Figure 3 a picture of assembled system is shown. The gap is always around 100 μm for both devices.

The loss angle measurement is made by ring down method exciting normal modes at their own resonant frequency by a piezoelectric actuator which is fixed on the same samples floor. With this method we measured Silicon and SiC loss angle in the 2-300 K temperature range.

The measured Silicon disk is 0.5mm in thickness and 10cm in diameter. We measure the loss angle of three normal modes (380, 1730 and 2510 Hz). Frequency refers to room temperature because for each mode the resonance frequency is a function of temperature. The frequency values are in agreement with FEM analysis within 5%. In Figure 4 the measurements are reported. The continuous line is the prediction of thin disk thermoelastic dissipation model due to Ferreirinho [6, page 130 and therein]. The thermoelastic dissipation seems to be dominant above 40K where all other contributions seems to be negligible. The lowest measured loss angle is $\phi = 1.35 \times 10^{-8} @ \sim 7K$.

Given the uniform thickness $t$ and the radius $r$, in the hypothesis of free edge, the mode resonant frequency $f_n$ for a free homogeneous disk is given by

$$f_n = \frac{k_n}{2\pi} \sqrt{\frac{D\gamma}{\omega r^4}} \quad (2)$$

where $D = ET^2/12(1-\nu^2)$, $\nu$ is the Poisson’s ratio, $\omega$ the uniform load per unity area included the own disk weight, $E$ the elasticity modulus and $k_n$ a constant depending on the considered normal mode [7]. Since the temperature dependence of all the other parameters is negligible, Young modulus, $E(T)$, determines the temperature dependence of the resonant frequency. Furthermore, since the suspension is nodal, the hypothesis of free disk can be considered satisfied. $E(T)$ can be calculated using the semi-empirical formula [8]

$$E(T) = E_0 - BTe^{-\frac{T_0}{T}} \quad (3)$$
Figure 2. Sketch of the nodal suspension realized on thin disks. The clamping spheres are visible in green (steel for SiC and sapphire for Si). Here both optical lever and capacitive readout are shown. On bottom a sketch of the displacement sensor for dielectrics is shown.

Figure 3. Silicon thin flat plate (thickness = 0.5 mm) is housed on nodal clamping mounting. It is visible the support for the dielectric capacitive readout. On bottom left a picture of the multistrip displacement sensor used is shown.

Figure 4. Silicon loss angle measured on thin flat plate (thickness = 0.5mm) as function of temperature. Continuous lines represent the thermoelastic dissipation according to Ferreirinho (more details in the text).

Figure 5. Silicon Carbide loss angle measured on 1mm thickness flat plate as function of temperature.

where $E_0$ is the Young Modulus at 0K, $B$ is the bulk modulus, $T_0$ is related to the Debye temperature and $T$ is the temperature in Kelvin. With this model it is possible to fit $f(T)$ data to obtain $E_0$, $B$ and $T_0$. In Figure 6 two examples of fit are shown. The fitted values agree for all the normal modes investigated and on average are $E_0 = 167.5 \pm 0.5 GPa$, $B = 15.80 \pm 0.03 MPa$, \ldots
\( T_0 = 310 \pm 8 K \). In Figure 6 an example of fit is shown.

**Figure 6.** Frequency as function of temperature measurements. The continuous red lines represent the fit with the model described in the text. The missing points at high temperature are probably due to a bad thermometer calibration. New measurements and thermometer calibration are yet scheduled for the next run.

We measured SiC loss angle in two disks of 5 cm in diameter and 1 and 2 mm in thickness. The lowest loss angle measurement is \( \sim 3 \times 10^{-6} \) @ 2 K. For SiC the loss angle increases regularly with the temperature. The measurements on 1mm thickness SiC plate are shown in Figure 5.

5. **Future perspectives on loss angle measurements.**

The measurements of Silicon loss angle show \( T' \phi \simeq 9 \times 10^{-8} K \). This value is not too far from DUAL requirement of \( T' \phi \lesssim 10^{-8} K \) and agrees with measurements in literature. The next step will be the measurement of the dissipation of direct silica bonding. The first sample is machined and is ready for measurement. It was obtained bonding together three identical P-doped silicon disks 0.5mm in thickness and 10cm in diameter.

Investigations on SiC will continue at ultra low temperature in order to verify if there is a loss angle improvement. In the next future investigations on loss angle of Mono-Crystalline Silicon direct bonding are scheduled. Investigations on Silicon will continue for disks of different thicknesses to evaluate the volume/area ratio effect on mechanical dissipation. Molybdenum is an interesting material as shows low dissipation at low temperature; measurements of its loss angle at low temperature on disks will come soon.

The ”multistrip” capacitive readout for dielectric samples was employed in a cryogenic run for the first time with good results. Researches and studies about it will continue in order to improve displacement sensor sensitivity. On the other hand the ”multistrip” sensitive area was optimized varying the geometrical parameters in order to maximize the displacement sensitivity and minimizing the sensitive area. The first sensor is yet ready and will be tested in the next cryogenic run. Further improvements are possible in the next future, trying to achieve a thermal noise sensitive readout.

[7] Roark, Roark’s formulas for stress and strain