Resistivity Characterization of Molybdenum-Coated Graphite-Based Substrates for High-Luminosity LHC Collimators

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Abstract: The High-Luminosity Large Hadron Collider (HL-LHC) project aims at extending the operability of the LHC by another decade and increasing by more than a factor of ten the integrated luminosity that the LHC will have collected by the end of Run 3. This will require doubling the beam intensity and reducing the transverse beam size compared to those of the LHC design. The higher beam brightness poses new challenges for machine safety, due to the large energy of 700 MJ stored in the beams, and for beam stability, mainly due to the collimator contribution to the total LHC beam coupling impedance. A rich research program was therefore started to identify suitable materials and collimator designs, not only fulfilling impedance reduction requirements but also granting adequate beam-cleaning and robustness against failures. The use of thin molybdenum coatings on a molybdenum–graphite substrate has been identified as the most promising solution to meet both collimation and impedance requirements, and it is now the baseline choice of the HL-LHC project. In this work we present the main results of the coating characterization, in particular addressing the impact of coating microstructure on the electrical resistivity with different techniques, from Direct Current (DC) to GHz frequency range.

Keywords: molybdenum coatings; MoGr; graphite; resistivity; grain size; microstructure; collimators; LHC; HL-LHC; beam stability; impedance; SEM; FIB; eddy current; four-probe; cavity

1. Introduction

The LHC was designed with a very efficient and robust 3-stage collimation system [1–3] put in place to protect the accelerator against regular and accidental beam losses. The system ensures adequate beam-cleaning keeping energy deposition due to losses in the superconducting magnets below quench limits, and background at experimental detectors and losses at sensitive equipment at tolerable levels. It consists of over 100 collimators, with two movable jaws that are aligned symmetrically around the beam [4].
As shown in Figure 1 the system is mainly located in two dedicated Insertion Regions (IRs) according to their specific purposes: IR7 for betatron cleaning (e.g., machine protection against transverse losses) and IR3 for momentum cleaning (e.g., cleaning of un-captured beam at the beginning of the energy ramp, or machine protection against radio frequency (RF) failures) [5]. Also, injection protection devices are installed in IR2 and IR8, as well as extraction protection devices are installed in IR6, to protect the machine against injection and extraction kicker mis-firings. Additional collimators are installed for local protection of the final focusing system, for reduction of the background to the detectors at the experimental interaction points and for cleaning of collision products that have magnetic rigidities similar to the circulating beams.

The system is organized in multiple stages. Target Primary Collimators (TCPs) constitute the smallest bottleneck of the ring and intercept large-amplitude halo protons. Secondary collimators (also referred to as TCSGs, Target Secondary Collimators Graphite) catch the secondary halo leaking out of the TCPs. Active absorbers (also referred to as Target Collimator Long Absorbers or TCLAs) attenuate particle showers and intercept a part of the tertiary halo coming out of TCSGs. Tertiary collimators (also referred to as Target Collimator Tertiary or TCTs) in the experimental insertions protect the local aperture bottlenecks and help controlling the experimental backgrounds [6,7].

Figure 1. Schematic representation of the LHC collimation system during Run 2 (2015–2018) [8].

When a particle beam passes near a collimator jaw it induces electromagnetic wake fields, which can in turn act back on the beam, distorting its motion. The strength of this interaction is represented by the beam coupling impedance (also referred to as impedance) [9,10]. The jaws of primary and secondary collimators are located close (1–3 mm) to the beam and need to be robust against beam impacts. In the present LHC they are therefore made of Carbon Fiber Composite (CFC) and, due
to their proximity to the beam and high material resistivity, they represent a significant source of impedance of the LHC. The tertiary collimators and absorbers are made of a tungsten alloy, specifically chosen for an increased absorption at the price of robustness, and usually operated at larger apertures, thus contributing less to impedance. Overall, the collimation system currently represents the largest source of transverse impedance in the LHC at 6.5 TeV as shown in Figure 2 for the horizontal plane over a wide frequency range [11]. In particular, the IR7 collimators, due to their small gaps, constitute the single largest contribution to the total LHC impedance.

Interaction with the beam coupling impedance can induce coherent instabilities in the beam if mitigation means are not actively put in place. In the LHC, the beam stability is currently ensured by the octupole magnets and by the transverse feedback. The former induces a spread in betatron frequency proportional to particle amplitude to provide Landau damping to all impedance-driven unstable modes [12], while the latter provides active bunch-by-bunch correction kicks that damps unstable low-frequency coherent coupled-bunch oscillations. The most critical of the two systems, in view of the beam parameters foreseen by the HL-LHC project [13], are the octupole magnets, which are limited to a maximum current of 570 A and hence provide a maximum amount of betatron spread.

The upgrade of the machine foreseen by HL-LHC poses new stability challenges brought by a higher beam brightness, in particular for the stability of the most challenging baseline type of beam, the so-called BCMS [14] achieved with Batch Compression, Bunch Merging and Splitting in the CERN PS accelerator: $2.3 \times 10^{11}$ p per bunch versus $1.2 \times 10^{11}$ p per bunch and a slightly, $\sim$15% smaller transverse emittance (proportional to the square of the rms beam size at any given location, for reference see for example [15]) compared to that of LHC Run 2 (Table 1). In this frame, the capabilities of the octupole system will not be sufficient to grant beam stability in the most challenging operational scenarios. Given the collimation contribution to the total machine impedance, the main mitigation measure is to reduce the collimator impedance by lowering the resistivity of the jaws with alternative substrate materials and coatings. For this reason, we extensively investigate the possible alternatives and characterize their electrical resistivity to meet the required beam stability target. In particular, we
address the impact of the coating microstructure on the final electrical resistivity and the relation to the specific coating process and substrate characteristics.

Table 1. LHC design, LHC Run 2 and HL-LHC parameters for the most critical beam (BCMS [14]).

<table>
<thead>
<tr>
<th>Parameter</th>
<th>LHC Design</th>
<th>LHC Run 2</th>
<th>HL-LHC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beam energy (TeV)</td>
<td>7</td>
<td>6.5</td>
<td>7</td>
</tr>
<tr>
<td>Bunch intensity (10^{11})p</td>
<td>1.15</td>
<td>1.2</td>
<td>2.3</td>
</tr>
<tr>
<td>Number of bunches</td>
<td>2808</td>
<td>2556</td>
<td>2748</td>
</tr>
<tr>
<td>Normalized emittance</td>
<td>3.5 (\mu)m</td>
<td>2.5 (\mu)m</td>
<td>2.1 (\mu)m</td>
</tr>
</tbody>
</table>

In Section 2 we present the upgrade plans for the collimator impedance reduction in LHC and the final choice of materials to be studied as a low-impedance alternative for the HL-LHC upgrade, including thin molybdenum coatings of collimator jaws. In Section 3 we describe the sputtering techniques used to prepare the coatings. In Section 4 we show the results of laboratory measurements of coating resistivity obtained by various complementary techniques: the four-probe measurement at DC, eddy current testing at low frequencies, resonant cavity measurement at microwave frequencies. We then discuss in Section 5 the impact of the coating procedure on its microstructure and consequently on its performance as an impedance mitigation measure. We conclude with the present status and plans for coating fabrication for the ongoing LHC collimator replacement.

2. Considered Material Choices for Collimation

The absorber material of collimators often exploits graphite-based materials for their low atomic number and excellent thermal properties, which ensures mechanical robustness in accident scenarios [16]. Presently, CFC (Tatsuno AC150K grade [17]) is used in primary and secondary collimators. This is an anisotropic material with an in-plane DC resistivity of about 5 \(\mu\)Ohm. The choice of alternative materials to replace CFC has been a field of intense research in recent years [13]. A family of novel graphite-based composites reinforced with a dispersion of molybdenum-carbide particles, referred to as Molybdenum-Carbide Graphite (MoGr), with very high thermal and electrical properties, was recently developed at CERN, in collaboration with Brevetti Bizz (IT), for the upgraded primary and secondary collimators. This material, produced by spark plasma sintering, assisted by liquid phase, has been extensively investigated and characterized [18].

The in-plane DC resistivity \(\rho\) used for beam stability computations is shown in Table 2 with references therein. The reader must note that the CFC resistivity reported in the table is corresponding to the grade of the jaw material presently installed in the LHC (i.e., AC150K), whose production has been discontinued by the company. In this study we use a new grade, Tatsuno FS140K, which has slightly higher resistivity than the previous one although both grades are graphitized at 2800 °C [17]. MoGr electrical conductivity is higher than that of CFC by a factor of about 5. In the case of IR7 secondary collimators, the selected material (MoGr grade Nanoker NB-8304Ng [19]), will also be coated with a thin metallic layer, further boosting the surface electrical conductivity. Primaries in the same IR cannot be coated as the higher beam induced losses would vaporize the coating in a few turns. Apart from low resistivity, the coating material must show good adhesion to the bulk substrate and have a large melting point to prevent damage against beam losses. In order to verify the coating robustness, different graphitic materials coated with metallic thin films have been tested in the CERN HiRadMat facility [20] with proton beams. Among the proposed metallic thin films, the molybdenum coating shows the least extended beam damage, mainly due to its higher melting temperature. Based on the outcome of these studies, a 5 \(\mu\)m thick metallic molybdenum coating has been chosen, which improves the surface electrical conductivity by an additional order of magnitude, as reported in Table 2. The thickness of the coating is such that it effectively screens the bulk of the jaw in the range of frequencies above 400 MHz, which is relevant for single bunch beam dynamics in
LHC [13], provided that a sufficiently strong feedback system suppresses the low-frequency unstable oscillations in the beam. It is important to underline that the coating is applied on the in-plane surface of MoGr, the electrical resistivity of which is about 5 times lower than that of CFC. In order to keep the resistivity low in case of partial detachment of the coating, it is preferable to apply a coating on MoGr rather than CFC. As a comparison, we decided also to investigate Mo coating layers on other graphitic materials involved in the collimation system, such as CFC and isotropic polycrystalline graphite (SGL R4550 and R7550 equivalent grades [21]).

Table 2. Summary of CFC, graphite, MoGr and Mo electrical resistivity at DC assumed in beam stability computations (for anisotropic materials the resistivity is considered on the beam direction).

<table>
<thead>
<tr>
<th>Material</th>
<th>CFC</th>
<th>Graphite</th>
<th>MoGr</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\rho$ [n(\Omega)m]</td>
<td>5000 [22] 1</td>
<td>13,000 [21]</td>
<td>1390 2</td>
<td>53.4 [23]</td>
</tr>
</tbody>
</table>

1 This value corresponds to the grade of material of the jaws presently installed in the LHC (i.e., AC150K), which is lower than that used through the present paper (i.e., Tatsuno FS140K). 2 This value is derived from the analysis of the samples measured in this paper as reported in Section 4. For collimator jaws, it has been observed [22] a decrease of the resistivity close to the surface of about 15% (1170 n\(\Omega\)m) related to the production process. Nevertheless, for stability computations we adopted the most pessimistic one.

Let us now consider the impact on beam stability from the different material choices in the upgrade of the IR7 collimators. Figure 3 shows the present octupole current required in HL-LHC, in case no collimator upgrade takes place (violet bar). Based on the present operational experience, the octupole current has to exceed the limit defined by an ideal impedance model by about a factor two in order to account for a variety of effects ranging from imperfections of beam optics to hardware noise, to interaction between the two counter-rotating beams [24]. With that in mind, the maximum allowed octupole current of 570 A would be exceeded with the existing collimation system. In order to solve this problem, the HL-LHC project baseline foresees that two out of four primary collimators per beam will be upgraded from CFC to MoGr, and 9 out of 11 secondary collimators per beam, which contribute the most to the overall beam coupling impedance, will be replaced with new collimators that have Mo-coated MoGr jaws. If all secondary collimators were upgraded to MoGr only (i.e., without low-impedance coating), as the primaries, the required stabilizing octupole current would be lowered by \(\sim 200\) A (yellow bar). The baseline of Mo coating on 9 out of 11 secondary collimators further reduces the required octupole current (red bar). Please note that in this paper, for the sake of simplicity, we only focus on stability of a single beam. In practice long-range interactions of counter-rotating beams could further reduce the required stabilizing octupole current below 570 A in the HL-LHC baseline scenario [24].

The coating has been considered on a variety of substrates: CFC (FS140K), MoGr (NB-8304Ng), and graphite (SGL R4550/R7550). While the resistivity of the substrate is not important for beam stability, provided that the coating thickness is sufficiently large, studies show that a good adhesion and a low coating resistivity is achieved when using MoGr as substrate material (see Section 4 for details). Alternative coatings such as Cu on CFC or MoGr were also studied in dedicated HiRadMat tests [25] but ultimately deemed unfavorable due to the high risk of radiation damage induced in the coating due to beam scratching or impact. For this reason, we restrict ourselves to the analysis of Mo coating layers.
3. Sputtering Coating Techniques

Different sputtering techniques were used to deposit thin Mo coating layers on the CFC, MoGr and graphite substrates described in the previous section. Prior to the full collimator production, small samples were studied to assess the impact of the different techniques on the electrical resistivity. In order to guarantee a good adherence and reduce outgassing, the samples were prepared before the coating deposition with the following steps:

- cleaning in ethanol bath with ultrasonic agitation for $2 \times 15$ min;
- drying in an air oven at $60^\circ$C for at least 1 h;
- vacuum-firing at $950^\circ$C at $10^{-6}$ mbar for at least two hours to reduce outgassing.

The Mo coating on the MoGr samples is performed by Direct Current Magnetron Sputtering (DCMS) [27] and by High-Power Impulse Magnetron Sputtering (HIPIMS) [28]. In the case of DCMS, samples are coated at CERN with temperature in the range of 250–300°C. They are loaded on a rotating support at a distance of 100 mm above two semi-balanced magnetron sources (GENCOA UH-012; Ø 150 mm) with Mo targets (99.95 % purity). Prior to coating, the vacuum chamber is pumped down and a bake-out at 135°C during 24 h is applied. The base pressure, reached prior to coating, is in the low $10^{-8}$ mbar range. 6 µm thickness of Mo (i.e., slightly larger than the required 5 µm) is deposited in 7 h with Krypton process gas and with $2 \times 300$ W power.

In the case of HIPIMS, samples are coated by DTI [29] in a standard batch coater with a satellite rotating system and Mo targets (99.95%) with temperature in the range of 300°C. 6 µm thickness of Mo was deposited in 3.5 h with Krypton process gas and firm specific HIPIMS process parameters. Due to the large material porosity and consequently large electrical resistivity measured for DCMS coated CFC samples, tests with HIPIMS were not done for this type of substrate (see Section 4 for details).

The sample thickness is measured by calotest and by a stylus profilometer. Adhesion of the coating was tested in a pull test and showed no delamination of the Mo coating. In particular, a rupture in the MoGr planes occurred at around 6 MPa, but never at the interface between the Mo coating and the MoGr substrate [30], which is close to the through-plane strength of the bulk material [18].
4. Resistivity Measurement Techniques

An accurate estimation of the electrical resistivity of MoGr and graphite bulk substrates and of Mo coating layers is of high importance in order to correctly predict the stability threshold of Figure 3. To qualify the resistivity of Mo coating layers and MoGr or graphite substrates, different techniques can be used. In the following we will describe different approaches ranging from DC to low frequencies (from a few kHz to MHz range) and to high frequencies (in the GHz range).

4.1. Two-Probe and Four-Probe Methods

The resistivity of a homogeneous conductor of constant transverse section is given by second Ohm’s law as

$$\rho = \frac{R A}{d}$$

where $R$ is the measured resistance, $A$ the cross-section area, and $d$ the length of the sample.

In the two-probe (or two-point) method [31], we directly measure the resistance with a multimeter and two probes are placed at the sample extremities. We therefore include the contact resistance between the sample and the electrodes, which needs to be taken into account in the estimate of the sample resistance. This limitation can be overcome by the four-probe (or four-point) method, in which the current is measured at the sample extremities with an ammeter, and the voltage drop across the inner part of the sample with a voltmeter. In this case, the resistivity becomes

$$\rho = \frac{V}{l} \frac{w d}{d}$$

where $d$ is the distance between the voltage probes, $w$ the width of the sample and $t$ its thickness.

The custom-built measurement setup is analogous to that proposed by the standard [32], composed by a power supply connected to two copper electrodes and a voltmeter connected to two probes. In order to improve the electrical contact with the sample, a soft and conductive material (adhesive electromagnetic shielding gasket) is placed on the copper electrodes.

For sufficiently thick samples (usually of about a few mm in thickness), the current can be applied between parallel surfaces, as shown in Figure 4. For thin films a different design is used, applying the electrodes directly on the top of the coated surfaces, as shown in Figure 5. The two electrodes are attached to an insulating support to hold them at a fixed distance.

![Figure 4. Scheme of the four-probe setup used for bulk measurements.](image-url)
Figure 5. Scheme of the four-probe setup used for thin film measurements.

For bulk samples, the electrical bulk resistivity measured with the scheme of Figure 4 is summarized in Table 3.

Table 3. Electrical resistivity of CFC, graphite and MoGr measured with the four-probe method on thick samples. $X$, $Y$, $Z$ subscripts represent the 3 orthotropic directions of the materials (only graphite is isotropic).

<table>
<thead>
<tr>
<th>Material</th>
<th>$\rho_X$ (nΩm)</th>
<th>$\rho_Y$ (nΩm)</th>
<th>$\rho_Z$ (nΩm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CFC</td>
<td>32,600</td>
<td>8200</td>
<td>5700</td>
</tr>
<tr>
<td>Graphite</td>
<td>12,600</td>
<td>12,600</td>
<td>12,600</td>
</tr>
<tr>
<td>MoGr</td>
<td>14,200</td>
<td>1390</td>
<td>1390</td>
</tr>
</tbody>
</table>

Once the material is installed in a collimator jaw, we consider $\rho_Y$ and $\rho_Z$ the resistivities in the in-plane directions that form the jaw active surface ($\rho_Z$ is the one parallel to the beam trajectory), and $\rho_X$ the resistivity in the direction perpendicular to it.

For thin films, the measured resistance can be extremely high due to the very small transverse cross-section. For this reason, if we want to measure their resistivity we need an insulating substrate bulk below the coating. In this way, the preferred current flow is through the coating, and therefore the measured voltage drop is dominated by the coating resistivity. The collimator jaw materials, and especially MoGr, are conductive. For this reason, if we consider a bulk with a thickness of a few mm, the current would flow through it affecting the estimate for the coating resistivity. Therefore, it was decided to produce very thin bulk samples, with a thickness of about 150 µm. By decreasing the size of the available bulk section for the current flow and by considering the higher electrical conductivity of the coating material, we obtained a comparable resistance between the two media. A picture of the experimental setup is reported in Figure 6. When measuring, the sample is placed between the setup and a flat surface. In particular, the shielding gasket is in contact with the sample and with the electrodes. All the system is screwed to an insulating support that is open in the middle to allow the voltage measurement.

A comparison of the resistance ratio between Mo and MoGr obtained for the two bulk thickness cases is shown in Table 4.
Table 4. Ratio of Mo coating to MoGr bulk resistance in function of the bulk thickness. The MoGr resistivity considered is shown in Table 3.

<table>
<thead>
<tr>
<th>Thickness (mm)</th>
<th>$R_{\text{bulk}}$ (mΩ)</th>
<th>$R_{\text{coating}}/R_{\text{bulk}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.28</td>
<td>33</td>
</tr>
<tr>
<td>0.15</td>
<td>9.3</td>
<td>1</td>
</tr>
</tbody>
</table>

With a 0.15 mm-thick bulk, we can consider a parallel resistance model in which the contribution of both layers is relevant. By knowing the resistivity of the bulk $R_{\text{bulk}}$ and by measuring the resistance of the coated sample, $R_{\text{parallel}}$, it is, therefore, possible to obtain the coating resistance $R_{\text{coating}}$ using the following formula

$$R_{\text{coating}} = \frac{R_{\text{bulk}} R_{\text{parallel}}}{R_{\text{bulk}} - R_{\text{parallel}}}$$

(3)

From Equation (2), the coating resistivity $\rho_{\text{coating}}$ is

$$\rho_{\text{coating}} = R_{\text{coating}} \frac{w}{t_{\text{coating}}}$$

with $t_{\text{coating}}$ the coating thickness.

Even if the resistivity of the bulk is well known by means of other methods, the in-plane resistivity of each sample has been measured before coating, in order to minimize the error induced by scattering of bulk resistivity. The thickness of the samples is measured with a non-contact method, with a micro-metric resolution. A summary of the values found for the measured substrates is reported in Table 5.

Table 5. Resistivity of CFC, graphite and MoGr measured with the four-probes method on thin samples.

<table>
<thead>
<tr>
<th>Material</th>
<th>CFC</th>
<th>Graphite</th>
<th>MoGr</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\rho$ (nΩm)</td>
<td>9900 ± 3000</td>
<td>9900 ± 3100</td>
<td>1240 ± 330</td>
</tr>
</tbody>
</table>

The measurements are then repeated for the coated samples. In particular, we report in Figure 7 the measurements done on DCMS Mo coating on MoGr, CFC and graphite and HIPIMS Mo coating on MoGr and graphite. As we can see from the graph, the DCMS technique gives different results for the different substrates: the resistivity of the coating on MoGr is four times lower than that on CFC or graphite. If we compare the HIPIMS Mo coating on graphite and MoGr we see that with this technique the resistivity reaches the theoretical value for pure Mo, lower with respect to DCMS in both cases. The error bars refer to the combined uncertainty calculated from the individual uncertainties of each of the measured parameters (instruments resolution) [33]. The reason for the high resistivity of Mo on CFC has been immediately associated with the intrinsic millimetric-size porosity present on the material surface. For this reason, the HIPIMS technique was not applied on it. The understanding of the difference between MoGr and graphite substrates and the impact of DCMS and HIPIMS techniques on the final coating resistivity is subject of a detailed discussion on the microscopic aspects in Section 5.
Figure 7. Comparison of measured resistivity of Mo coating layers produced with HIPMS and DCMS techniques on graphite, MoGr and CFC substrates.

4.2. Eddy Current Testing

Eddy Current Testing (ECT) is one of the electromagnetic methods used in non-destructive tests making use of electromagnetic induction to detect and characterize surface and sub-surface defects in conductive materials [34]. The principle of the eddy current technique is based on the interaction between a magnetic field source and a test material. When an alternating current flows through a coil, it generates a primary electromagnetic field. For frequencies below the self-resonance of the coil, the coil can be seen as an inductor generating a primary magnetic field. When the field is approached by an electrically conductive material, the primary magnetic field induces eddy currents which generate in turn a secondary magnetic field as shown schematically in Figure 8. As a result, the measured coil input impedance $Z$ will change by an amount $\Delta Z$ according to the electromagnetic properties of the material under test.

$\delta = \sqrt{\frac{\rho}{\pi \mu_0 f}}$  

(5)
with $\mu_0$ the vacuum permeability. When the thickness of the materials is typically less than 3 times the skin depth $\delta$, these devices cannot be used in a straightforward manner but a complementary approach can be adopted as described in this section.

The input impedance for a coil suspended at height $h$ (also known as lift-off parameter) on a multilayered, infinitely wide, linear, homogeneous and isotropic medium was studied in [35], extending the work of [36] previously done for one layer on an infinite substrate. Figure 9 schematically shows the procedure adopted for measuring coating and bulk resistivity. To measure the bulk resistivity and the lift-off parameter, the coated sample can be put in configuration B (i.e., with coating far from the coil) on a common and thick substrate (e.g., a slab of copper) and compared to configuration C (i.e., with respect to the measurement taken on the substrate only); to measure the coating resistivity, once the bulk is known, the sample can be measured in configuration A versus B. Measurements are then compared, in a least-square sense, to simulations performed implementing [35], from which the lift-off, bulk and coating resistivity can be deduced.

Figure 9. ECT schematic procedure for resistivity measurements. Measurements in configurations B versus C give information on bulk resistivity and lift-off $h$; the measurement in configuration A versus B gives information on the coating resistivity. The thickness of the $N$-turns coil, of the coating and the bulk are also shown. The configuration C is taken with respect to the common substrate.

Figure 10 shows the measurement setup. A coil is connected to an impedance analyzer working from 20 Hz to 2 MHz and suspended on the sample under test. The coil is made of 19 turns, with an inner radius of 1.58 mm, outer radius of 1.86 mm, and an overall thickness of 12 mm.

Figure 10. ECT setup. The impedance analyzer is connected to a coil suspended over a sample under test.

4.2.1. Application to Bulk Materials

Let us consider first an application of the method to the determination of the resistivity of bulk materials. This implies the application of the procedure shown in Figure 9 in configurations B and C; the coil input impedance measured in configuration C is subtracted from the one in configuration B, and compared with simulations to assess the bulk resistivity and lift-off parameter. We let the reader notice that the presence (or not) of a coating layer, while in configuration B, is not significantly affecting the coil input impedance if the thickness of the bulk is sufficiently larger than that of the coating.

Figure 11 shows the measured coil input impedance change $\Delta Z = \Delta R + j\Delta X$, together with simulated response for a graphite sample of $t_{\text{Bulk}} = 1.5$ mm placed on a thick (10 mm) copper substrate at a lift-off of $h = 0.7$ mm from the coil. The frequency ranges from 100 kHz to 2 MHz in steps of
100 kHz for 5 different positions along the center of the samples to collect statistics on the material homogeneity. We let the reader notice that the coil response increases in magnitude with frequency both for the real and imaginary part of $\Delta Z$ (marked by the black arrow).

Figure 11. Response of the coil on a graphite substrate of 1.5 mm thickness and $h = 0.7$ mm lift-off (measured data with dashed lines representing the measurement standard deviation) compared to different simulated responses varying the bulk resistivity between 5000 n$\Omega$m and 15,000 n$\Omega$m. The lift-off parameter $h$ can be inferred by matching the position of the data-point corresponding to the maximal acquired frequency $f_{\text{max}}$ (2 MHz in our case) to the curves obtained computing $\Delta Z$ for different lift-off parameters and $\rho_{\text{bulk}}$, as shown in Figure 12.

Figure 12. Response of the coil on a graphite substrate for the maximum acquisition frequency of $f_{\text{max}} = 2$ MHz with simulated responses for different lift-off parameters $h$ and bulk resistivity between 5000 n$\Omega$m and 15,000 n$\Omega$m. The intersection of the curves with the data-point gives the actual lift-off of the measurement, i.e., $h = 0.7$ mm.

By looking at the minimum distance between $|\Delta Z|$ and the simulated response for various bulk resistivity values, one can infer the resistivity of the sample under study. Figure 13 summarizes the
measured resistivity for CFC, graphite and MoGr samples. It is interesting to notice that the resistivity of graphite and CFC samples, despite the large penetration depth in the material, can be obtained already for $t_{bulk}/\delta \simeq 0.5$. Table 6 summarizes the measured bulk resistivities by averaging the values obtained for frequencies in which $t_{bulk}/\delta > 0.5$, compatible within errorbars to the measurements done at DC in Table 5.

![Figure 13. Measured resistivity for CFC, graphite and MoGr samples as a function of frequency (dotted lines) together with sample thickness normalized to the material penetration depth (dashed lines).](image)

<table>
<thead>
<tr>
<th>Material</th>
<th>CFC</th>
<th>Graphite</th>
<th>MoGr</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\rho$ (n$\Omega$m)</td>
<td>7497 ± 13</td>
<td>12,640 ± 130</td>
<td>1380 ± 30</td>
</tr>
</tbody>
</table>

4.2.2. Application to Coatings

Once the lift-off and the bulk material resistivity are known, the procedure of Figure 9 can be applied. The coil input impedance measured in configuration B is subtracted from that from configuration A, and compared with simulations to assess the coating resistivity.

A similar method was proposed by [37], with the exception that the authors focused on a specific frequency span around the zero-crossing of $\Delta R$ which implied difficulties in the measurements of bulk materials resistivity. In this work, the full $\Delta Z$ samples over frequency have been compared in a least-squares sense to the simulations, which allowed the same inversion algorithm to be applied to coatings and bulk materials with no significant limitations.

Figure 14 shows the dependence of the coil response on the coating resistivity for a given bulk resistivity, while Figure 15 shows the effect of the bulk resistivity for a given coating resistivity. In both cases, the larger the ratio between coating and bulk resistivity $\rho_{coating}/\rho_{bulk}$, the larger the magnitude of $\Delta Z$ over frequency. It is important to notice also the magnitude of $\Delta Z$: while for the measurement of bulk resistivity this is in the order of a few dozen of m$\Omega$, for coatings, it strongly depends on the relative resistivity between coating layer and substrate, which can potentially pose challenges with respect to temperature effects, setup stability and bulk in-homogeneity. For example, a coating resistivity as high as $\rho_{coating} = 500$ n$\Omega$m on MoGr would give a response of less than a m$\Omega$ in inductance and fractions of m$\Omega$ in resistance (green curve of Figure 14).
Figure 14. Simulated coil response for variable coating resistivity as a function of frequency. We assume a bulk of 1.5 mm thickness, with a resistivity of 1000 nΩm, and a coating of 6 μm thickness. The lift-off is 0.7 mm.

Figure 15. Simulated coil response for variable bulk resistivity. We assume a bulk of 1.5 mm thickness, and a coating of 6 μm thickness with resistivity of 50 nΩm. The lift-off is 0.7 mm.

The procedure was used to characterize Mo-coated samples on MoGr and graphite substrates. Figures 16 and 17 show the measurement data and the curves obtained varying the coating resistivity as in Figure 14 respectively for graphite and MoGr samples coated with 6 μm of Mo made with HIPIMS.
Figure 16. Coil response for HIPIMS Mo coating on graphite. The measurements are shown with black lines together with standard deviation (dashed lines) and the simulated response for coating resistivity between 50 nΩm and 300 nΩm. The coating has a thickness of 6 µm, the bulk of 1.5 mm. The lift-off is 0.7 mm.

Figure 17. Coil response for HIPIMS Mo coating on MoGr. The measurements are shown with black lines together with the standard deviation (dashed lines) and the simulated response for coating resistivity between 50 nΩm and 300 nΩm. The coating has a thickness of 6 µm, the bulk of 1 mm. The lift-off is 0.7 mm.

Figures 16 and 17 show a systematic shift of the measured data-points with respect to the curves computed analytically for higher frequencies. This can be due to a systematic bulk resistivity difference while approaching the surface when in configuration A with respect to B. A more resistive bulk will right-shift the simulated curves and, as shown in Figure 15, the effect will be much larger for the real part than for the imaginary part of $\Delta Z$. Therefore, reconstructing the coating resistivity using the absolute value will reduce the effect of this systematic uncertainty. On the other hand, this issue could also be mitigated restricting the maximum frequency of the measurements to have $t_{bulk}/\delta \simeq 1$; in this way the fields would effectively penetrate the full sample in both configurations A and B (for MoGr it would imply stopping at 500 kHz, for graphite at 1.5 MHz as shown in Figure 13).
Figure 18 summarizes the measurements performed on MoGr and graphite for DCMS and HIPIMS techniques. The relative behavior of the measured resistivity is consistent with the one measured at DC, i.e., coatings made via the DCMS technique are systematically more resistive than those produced via HIPIMS. In particular, Mo-coated HIPIMS samples show resistivities close to that of pure Mo. The value of the measured resistivity is higher than that measured at DC, and a higher resistivity is found for HIPIMS on graphite with respect to MoGr. The difference may be related to the different temperature reached by the substrate during the deposition process on thin samples used for the DC measurements. The samples may become hotter during the coating and this could induce different grain growth and aggregation with respect to the thick samples used in ECT. In order to suppress the dependency on bulk-related effects, an alternative technique based on a cylindrical resonator working in $H_{011}$ mode has been applied as discussed in the next section.

![Figure 18](image)

**Figure 18.** ECT measurement of Mo resistivity on graphite and MoGr for DCMS and HIPIMS.

### 4.3. Resonant Cavity

A cylindrical resonator operating in the $H_{011}$ mode [38] could be used as an alternative method to the ECT. The basic principle is to leave one side of the resonator open so that different surfaces can be tested (e.g., coated samples). The choice of the mode of operation is dictated by the absence of current flows on the end cap extremities of the cavity, while the dimensions of the cavity are a compromise between the practical manufacturing and the high frequency of operation needed to have the skin depth smaller than the coating thickness. As an example, a cylindrical cavity with the diameter of 25 mm and height of 35 mm exhibits the $H_{011}$ (or $TE_{011}$) mode at 15.24 GHz, which has low current concentration on the end cap extremities as shown in the Eigenmode CST simulation [39] in Figure 19a. This resonant mode, in a cylindrical cavity, degenerates with an $E_{111}$ (or $TM_{111}$) which shows currents on the end cap and needs to be detuned. A possible detuning method consists of making the bottom plate 45° tapered which will largely affect the $E_{111}$ due to its high electric field concentration, while the $H_{011}$ will be slightly pushed to higher frequencies (16.6 GHz) due to the reduced volume as shown in Figure 19b.
Figure 19. $H_{011}$ mode magnetic field distributions in a cylinder cavity without tapers (a), and with taper (b).

Given the high frequency needed, a Vector Network Analyzer (VNA) ranging up to 50 GHz was used. Before starting the measurements, the VNA was calibrated using an electronic calibration kit around the frequency of operation of interest (i.e., 16.6 GHz). The resonator cavity was connected via coaxial cables to the VNA. Figure 20 shows the manufactured cavity connected to the excitation ports in which tiny loops couple to the magnetic field of the $H_{011}$ mode.

![Fabricated cylindrical resonator with removable end cap.](image)

Figure 20. Fabricated cylindrical resonator with removable end cap.

The unknown resistivity of materials can be obtained from the variation of $Q$ factor with respect to reference materials. In CST Eigenmode Solver the $Q$ factor was calculated for different end cap material resistivities. The variation of the $Q$ factor normalized to that of copper is shown in Figure 21. The curve is well reproduced by the relation

$$\frac{Q}{Q_{\text{ref}}} = \frac{a + b}{a\sqrt{x} + b}$$

where $x$ is the resistivity ratio of the measured and reference materials ($x = \rho / \rho_{\text{ref}}$), $a$ and $b$ respectively the power dissipated in the end cap and the rest of the cavity.
Figure 21. CST Eigenmode simulation of the $Q$ factor variation versus resistivity, normalized to copper.

Prior to the measurements, the VNA is calibrated to operate on $H_{011}$ mode resonant frequency; afterwards, calibration measurements are done on the copper end cap as a reference. The cavity is cleaned with ethanol before measuring each new sample. The sample under test is placed as the end cap on the top of the cavity. Lastly, a weight is used for stabilization. In order to keep the sample clean and to avoid surface contamination, a clean paper is used between sample and weight. These steps are repeated in the measurement of each sample. Figure 22 shows the application of the cavity measurement on Mo-coated graphite and MoGr samples prepared with DCMS and HIPIMS techniques. The black points represent the $Q$ factor measured for known samples with reference to copper. The points are therefore fitted by Equation (6) from which the resistivity of the unknown Mo samples is deduced.

Table 7 summarizes the measured resistivity achieved with the three described techniques for resistivity characterization of Mo coatings deposited on MoGr and graphite substrates with DCMS and HIPIMS. In addition to the relative agreement observed between ECT and DC measurements,
a good agreement is achieved between RF and ECT measurements.

Table 7. Resistivity (in nΩm) measured with the DC, ECT and RF techniques for DCMS and HIPIMS Mo coatings on MoGr or graphite substrates.

<table>
<thead>
<tr>
<th></th>
<th>Bulk</th>
<th>Mo Coating</th>
<th>DC</th>
<th>ECT</th>
<th>RF</th>
</tr>
</thead>
<tbody>
<tr>
<td>MoGr DCMS</td>
<td>120±64</td>
<td>440±80</td>
<td>470±37</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MoGr HIPIMS</td>
<td>47±21</td>
<td>68±4</td>
<td>61±2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Graphite DCMS</td>
<td>410±140</td>
<td>628±5</td>
<td>679±41</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Graphite HIPIMS</td>
<td>47±17</td>
<td>145±2</td>
<td>112±1</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

For completeness we also summarize the measurements of resistivity of bulk materials done with the resonant cavity. Given the higher frequency of operation, roughness effects become important. An accurate estimation can be done using the gradient model [40] with the rms roughness $R_q$ given in Table 8 [41] to correct the resistivity measured with ECT. The agreement is satisfactory and within the measurement uncertainty, as shown in Figure 23.

Table 8. In-plane electrical resistivity of CFC, graphite and MoGr measured with the resonant cavity, together with roughness $R_q$ parameter [41].

<table>
<thead>
<tr>
<th>Material</th>
<th>$\rho$ (nΩm)</th>
<th>Typical $R_q$ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CFC</td>
<td>14,400±1000</td>
<td>9.4</td>
</tr>
<tr>
<td>Graphite</td>
<td>13,700±1100</td>
<td>1.4</td>
</tr>
<tr>
<td>MoGr</td>
<td>2170±140</td>
<td>1.1</td>
</tr>
</tbody>
</table>

Figure 23. Measured resistivity with low-frequency ECT (purple bar) and high frequency RF (red bar) techniques compared with the resistivity obtained with ECT accounting for surface roughness with the gradient model (green bar).

5. Microstructure Observations

In the attempt to understand the different contribution of bulk and coating to the final conductive behavior, microstructural analyses were performed using a Zeiss Sigma field emission scanning electron microscope (SEM) and a Zeiss Crossbeam 540 focused ion beam-scanning electron microscope (FIB-SEM). All the observations presented in the following are performed on thick specimens only (at least 1.5 mm). No additional sample preparation was accomplished for the coating observations, as the samples are sufficiently conductive for SEM. The fracture surfaces are obtained by bending-rupturing the ensemble substrate-coating (coated side under tensile stress). For the characterization of the
porosity of bulk graphite-based materials, a Gatan Ilion II argon milling system was used to prepare the surfaces, because it allows the obtaining of larger polished areas than the FIB technique.

Figure 24 shows the SEM observations of a Mo-coated MoGr sample produced with DCMS. The fracture surface (Figure 24 bottom) shows grains with in-plane sizes up to 0.5 µm, and single grains typically extending over the whole coating thickness (~5 µm). The top-view of the coating clearly shows the in-plane grain size (Figure 24 top-right). Spheroidal clusters with sizes of about 5 µm protrude on the coated surface (Figure 24 top-left), which appear also for DCMS produced Mo coatings on other substrates such as CFC or graphite. Substrates with smaller roughness, such as glass, when coated in the same way do not show these clusters [41]. For this reason, their formation seems to be related to the substrate surface roughness affecting the grain growth.

Figure 25 shows the SEM observations of a Mo-coated MoGr sample produced with HIPIMS. One can notice how the grain separation is barely visible on the surface, whereas the grain dimension is roughly similar to that obtained with the DCMS technique. The coating surface is smooth, showing randomly oriented bladed structures (Figure 25 at the bottom-right).

![Figure 24](image1.png)

**Figure 24.** SEM observations of a Mo-coated MoGr sample produced with DCMS. The fracture surface (bottom) and the coating surface (top) are shown.

![Figure 25](image2.png)

**Figure 25.** SEM observations of a Mo-coated MoGr sample produced with HIPIMS. The fracture surface (bottom) and the coating surface (top) are shown.
Figures 26 and 27 show the Mo thin films deposited on graphite respectively with DCMS and HIPIMS. The two different coatings on graphite show similar characteristics than the respective ones on MoGr, and Mo grain dimensions are also comparable between these two substrates. However, the coatings on graphite show more discontinuities possibly linked to the surface morphology of the substrate.

![Figure 26. SEM observations of a Mo-coated graphite sample produced with DCMS. The fracture surface (bottom) and the coating surface (top) are shown.](image1)

![Figure 27. SEM observations of a Mo-coated graphite sample produced with HIPIMS. The fracture surface (bottom) and the coating surface (top) are shown.](image2)

Figure 28 depicts the cross-section microstructures of HIPIMS Mo coatings on graphite and MoGr, taken at smooth substrate areas. The comparison of the grain size, from image analysis of Figure 28, is shown in Table 9 and does not show considerable differences related to the substrates.
Figure 28. FIB cross-sections on Mo-coated samples produced on graphite (a) and on MoGr (b) substrates with HIPIMS. The contrast between grains is due to the electron channeling effect. Vertical lines are due to the FIB process.

Table 9. In-plane grain size of the coating cross-sections (average ± standard deviation) from image analysis of Figure 28.

<table>
<thead>
<tr>
<th>Location</th>
<th>on MoGr (µm)</th>
<th>on Graphite (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coating surface</td>
<td>0.29 ± 0.12</td>
<td>0.26 ± 0.11</td>
</tr>
<tr>
<td>Coating mid-thickness</td>
<td>0.24 ± 0.10</td>
<td>0.19 ± 0.08</td>
</tr>
</tbody>
</table>

From these observations, the grains of Mo coating layers on MoGr produced with DCMS appear poorly aggregated with respect to those produced with HIPIMS. The effect of the reduced grains connection can be modeled with a reduced transmission of the current flowing through the grain boundaries [42]. The model assumes columnar grains with boundaries located at an average distance $D$. The current flow is reduced at the boundary transitions according to the transmission parameter $T$, with values between 0 and 1. The increase in resistivity due to the grain boundaries $\rho_g$, with respect to the bulk substrate resistivity $\rho_0$ can be computed according to

$$\frac{\rho_0}{\rho_g} = 3 \left[ \frac{1}{3} - \frac{1}{2} \alpha + \alpha^2 - \alpha^3 \ln \left( 1 + \frac{1}{\alpha} \right) \right]$$  (7)

where

$$\alpha = \frac{\lambda_\infty}{D} \frac{1 - T}{T}$$  (8)

with $\lambda_\infty = 35$ nm the mean free path in molybdenum [43].

Figure 29 shows the relative increase in resistivity computed based on this model. Only assuming a very low transmission parameter the model can justify the observed resistivity increase of coating layers on MoGr produced with DCMS (here we consider the average grain size from Table 9). If the transmission parameter would be the same for HIPIMS samples, a factor 10 difference in grain size would explain the measured difference in resistivity values, which is not observed in Table 9. Therefore, mainly a large difference in the transmission parameter between DCMS and HIPIMS coatings could explain the measured resistivity values.
Figure 29. Relative change in resistivity with respect to the ratio of grain size $D$ and mean free path $\lambda_\infty$ for Mo coating layers. Full lines correspond to different transmission $T$ between grain boundaries. Dashed lines correspond to the mean RF values of Table 7.

To understand the worse resistivity of Mo coatings on graphite with respect to MoGr substrates, additional FIB observations were done on ion-polished surfaces. Figure 30 shows the main difference between MoGr (left) and graphite (right) substrates. Large µm-scale porosities are present on graphite which eventually translate into the in-homogeneity of the coating microstructure shown in Figures 26 and 27. Figure 31 shows the surfaces of HIPIMS Mo coatings on MoGr and graphite, demonstrating that the intrinsic substrate characteristics have a large influence on the final coating discontinuities. Eventually, this effect could be modeled as well, on average, as a lower transmission between grain boundaries as shown in Figure 29. For graphite, in summary, the HIPIMS technique only partially improves the resistivity of the coating with respect to the DCMS one, due to the additional porosity of the substrate, absent in MoGr.

Figure 30. SEM observations of ion-polished MoGr (a) and graphite (b) substrates. MoGr has its through-plane direction perpendicular to the image. Large µm-scale voids are visible on graphite (holes in black), while Mo-carbide inclusions are visible on MoGr (white spots).

Figure 31. SEM observations of Mo coating produced with HIPIMS on MoGr (a) and graphite (b). Note how the coating replicates the substrate features, producing discontinuities.
6. Conclusions

In this work we have summarized the resistivity measurements performed on Mo-coated samples of different graphitic materials foreseen or being considered for the impedance reduction program of the HL-LHC collimators.

We have characterized both bulk and coated materials with different techniques: the DC four-probes method, the low-frequency ECT, and RF resistivity measurements with a cylindrical resonator working in the H_{011} mode.

For the bulk measurements, the DC and ECT techniques are in good agreement. Measurements done at RF show higher resistivities which can be explained by the additional roughness effect. The increase in resistivity with respect to DC and ECT was quantitatively modeled by applying the gradient method with excellent agreement to observations.

For the coated samples measurements, we have observed a difference between the DC four-probe resistivity measurements done on thin (~150 µm) samples with respect to the ECT and RF done on thick ones (~1.5 mm). The DC values of coating resistivity are systematically lower than those measured with the other techniques. The difference may be related to the different temperature reached by the substrate during the deposition process. The thin samples may become hotter during the coating process and this could induce grain growth and aggregation with respect to the thick samples. On the other hand, a relatively good agreement between the three techniques has been shown when comparing DCMS and HIPIMS coatings, pointing to a structural difference of the two processes on the final coating resistivity. Transversely, applying these techniques on graphite or MoGr substrates produces coating with higher resistivity in the former with respect to the latter.

SEM/FIB observations were therefore performed to analyze the microstructure of Mo films deposited on MoGr or graphite with the two different deposition techniques. In the case of MoGr, the HIPIMS technique produces well aggregated grains, while grains are observed to be less aggregated when applying the DCMS technique. This can be qualitatively explained by a reduced transmission capability of the current flowing through the grain boundaries. The current flow is therefore better transmitted between the grains of HIPIMS Mo coatings with respect to DCMS deposition.

The SEM analysis also allowed to identify the large porosity present on graphite when compared to MoGr substrates. When these voids are exposed at the substrate surface, they can interfere during the film growing process, creating discontinuities between the grains which increase the final Mo resistivity. These big porosities are related to the production process of graphite; in fact, during the pyrolysis, the graphite precursors produce volatiles creating these voids.

The authors are also investigating another possible mechanism that could contribute to the increase of coating resistivity on graphite, i.e., the formation of Mo-carbide during the coating process. The lower conductivity of Mo carbides or their effect on the Mo lattice structure could affect the final value of resistivity as well.

In addition to the described techniques (DCMS and HIPIMS), recent RF resistivity measurements of Mo coatings on MoGr substrates produced at CERN in HIPIMS with a positive pulse (HIPIMS + PP) [44] showed values in line with those obtained in Table 7. The surface microstructure achieved with this particular technique is presently under study.

Based on these observations, we have demonstrated that the HIPIMS coating technique is superior to the DCMS one for Mo coatings on MoGr and resistivity close to the bulk value can be obtained. For this reason it is being used to produce Mo-coated MoGr secondary collimators for the HL-LHC project.

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**Conflicts of Interest:** The authors declare no conflict of interest.

### Abbreviations

The following abbreviations are used in this manuscript:

- **BCMS**  Batch Compression, Merging and Splitting
- **CFC**  Carbon Fiber Composite
- **ECT**  Eddy Current Testing
- **DC**  Direct Current
- **DCMS**  Direct Current Magnetron Sputtering
- **FIB**  Focused Ion Beam
- **HIPIMS**  High-Power Impulse Magnetron Sputtering
- **HIPIMS + PP**  HIPIMS with a Positive Pulse
- **HL-LHC**  High-Luminosity Large Hadron Collider
- **IR**  Insertion Region
- **LHC**  Large Hadron Collider
- **MoGr**  Molybdenum-Carbide Graphite
- **RF**  Radio Frequency
- **SEM**  Scanning Electron Microscope
- **TCLA**  Target Collimator Long Absorber
- **TCP**  Target Collimator Primary
- **TCSG**  Target Collimator Secondary Graphite
- **TCT**  Target Collimator Tertiary

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