Research article

Application of the mixing of the field of low amplitude AC bias with a strong field of linear slow varying to investigate the magnetic properties of materials

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ABSTRACT

The paper presents a new experimental method of the magnetic hysteresis loop investigation and the constructed measurement instrument. The principle of operation is to mix two magnetic fields: the first of these fields has a very low amplitude, a shape of sine wave (AC bias) and the frequency range order of $10^{-2}$ Hz. The second field has a very low frequency (order $10^{-2}$ Hz), a high amplitude and forms approximately a triangle wave. The presented, dedicated system is different in comparison to commonly used hystographs and it consists of coreless coils only. The measurement method gives the opportunity of obtaining more detailed measurements of the initial cycles in the material magnetizing process. In a special case, the magnetic field strength changes in the $B(H)$ curve can be stopped at any operating point and then it is possible to investigate local properties using AC bias field for various frequencies. The results revealed a successive displacement of the hysteresis loop and approaching to the steady state position.

1. Introduction

A hysteresis loop is the most frequently measured parameter to assess the magnetic properties of any investigated material. The basic and historical method, in a nutshell, is based on building a transformer with a core of the investigated material and simultaneous measurement of primary current and secondary voltage in the XY mode on an oscilloscope. Today's standard hystographs for industrial application have evolved from this transformer. There are widely available different types of hystographs in commerce. Some of them use more advanced magnetic field detectors (than secondary coil) e.g. a teslameter with a Hall-probe. A scientific application requires much more sophisticated devices. In the paper [1] the authors used X-ray beam and Compton scattering effect as a new tool measuring spin-specific magnetic hysteresis. The aim of the work [2] was the concept of a new method of testing the hysteresis cycles of ferromagnetic sheets with a thickness of up to 1.5 mm while the measurements were carried out under DC conditions. A hysteresis loop features depend not only on temperature of the investigated material and on magnetic field frequency but also on the radiation exposure. The authors in the paper [3] reviewed the research results of the radiation-induced demagnetization of permanent magnets. The surface area of the magnetic hysteresis loop and hyperthermia on magnetic nanoparticles have been studied in the paper [4] and it was necessary to construct a dedicated measuring system. Thus, there is no one universal measurement method for testing the magnetic hysteresis loop in any material. The new, just presented method does not use only one frequency of the alternating magnetic field in comparison to so far used methods, which 50 Hz or 60 Hz electrical networks are used. This method is based on mixing two fields: the first of these fields is sinusoidal (AC bias) and has a very low constant amplitude. Its frequency ranges from 10 Hz to $10^4$ Hz. The second field has a very low frequency (on the order of $10^{-2}$ Hz), high amplitude and forms approximately triangular wave, which allows to enter the saturation area in the $B(H)$ characteristic (magnetic flux density vs. magnetic field strength). Moreover, the method gives the opportunity to perform more detailed measurements in the initial cycles of the material magnetizing process. The slow triangle wave component changes between the magnetic field strength peaks $-H_{\text{max}}$ to and $H_{\text{max}}$ in the $B(H)$ characteristic and this wave can be stopped at any operating point. Then it is possible to investigate local properties using a low-amplitude AC bias field for different frequencies. The main reason of constructing the presented device is the need to study the magnetic properties of nanomaterials. An electron microscope image of a nanomaterial sample can show various structures – e.g. nanospheres or nanowires. There are gaps between nanoparticles that are filled with

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gases. It is possible to partially remove gases by grinding and pressing but this may change the physical properties of the nanomaterial. This is often undesirable. Consequently, forming the solid core (of shape like in a transformer etc.) from the nanomaterial being tested is practically impossible. Other commonly used hysteresis loop investigation methods are often difficult to apply. The presented method allows to examine a sample of various shapes and forms. In addition, more detailed researches have shown that this method is sensitive to the disadvantages of the materials tested, therefore it can be used for non-destructive magnetic inspection.

2. The measurement system and principle of operation

The setup contains a measurement module (Figure 1) and an electronic unit (Figure 3). There are supply and measurement coaxial solenoids in the module (Figures 1 and 2). The supply solenoid has 5300 turns, 1 mm wire diameter, 75 mm coil diameter inside, 108 mm coil diameter outside, 360 mm length, weight over 10 kg and 7.5 kHz resonant frequency. The measurement solenoid (sensing coil – pick-up coil) has 4800 turns, 0.08 mm wire diameter, 17 mm coil diameter inside, 98 mm length and resonant frequency of 26 kHz. The shape of a sample is a thin cuboid, but other shapes can get used. In this paper the sample has got dimensions of 0.4 × 10 × 200 mm. The tested material may have various forms. Sometimes it will be pressed powder. The device allows to make measurements on any solid sample or sample in a plastic cuvette that will fit inside the measuring coil. The sample should be shorter than the coil. The separated sample or material in the cuvette is located inside a plastic pipe with the wound measuring coil (Figure 2). Seems that this coil (4800 turns) is expected to be thick, but it should be noted that the cross-sectional area of the wire is 156 times smaller and has 5 times smaller average coil diameter in comparison to the cross-section of the supply coil. In effect, the pick-up coil has 4 layers, a winding thickness of less than 0.3 mm and a weight of over a several milligrams. Therefore, this coil is delicate, susceptible to mechanical damage and had to be covered with insulating tape.

The electronics unit (Figure 3) provides voltage signal \( u_s(t) \) to the supply coil and it processes signal from the measurement coil \( u_m(t) \). The microcontroller (Arduino Uno board, AVR ATmega328) drives the sine wave (AC bias) integral circuit generator (AD 9850) and a 12-bit resolution digital to analog converter (DAC). This DAC produces triangle wave signal (exactly a positive or a negative going staircase waveform). Next, the DAC signal is anti-quantization filtered and is shifted by half the distance between the maximum and minimum output voltage of the DAC. In effect, the DAC signal becomes symmetrical to the time axis. After that, the DAC signal and the AC bias are mixed and amplified.

The triangle wave signal in the supply coil causes a very slow magnetic field increase or decrease in a wide range magnetic field strength, which influences the magnetic properties of the investigated material. The time dependence of the supply coil voltage is given by approximately Eq. (1) and consists of two components (2). The first one is associated with AC bias signal and the second one is associated with DAC signal. The number \( n \) in (1) is a value continually written (by microprocessor program) to 12-bit DAC register.
where: $u_{k}$ – voltage on supply coil terminals, $a$ – amplitude of AC bias signal, $b$ – amplitude of slow triangle wave signal, $n$ – whole number written to DAC (by microcontroller) to generate triangle signal, $\omega$ – radial frequency of AC bias signal, $u_{AC}$ – sinusoidal (AC bias) voltage component, $U_{tr}$ – slow triangle wave voltage component, which is considered as a constant value in short time e.g. 100 ms.

The $n$ is a whole number and time-varying in the range 0…4095 periodically according to the algorithm: 2048, 2049…4094, 4095, 4094…2, 1, 0, 0, 1, 2…2046, 2047 and in effect after linearization the slow triangle wave is obtained. For each DAC set value, the electronics unit receives AC bias signal from the measurement coil. One full measurement cycle has 8192 measuring points. The values $n = 2047$ and $n = 2048$ correspond approximately to zero value of the static bias ($U_{tr}$) of the slow triangle wave is obtained. For each DAC set value, the electronics radial frequency of AC bias signal, $u_{AC}$ (4) is about 15 ms (inductance adjusted). Typical presets of coefficients $a$ and $b$ in Eqs. (1) and (3) are $a = 200$ mV and $b = 5.9$ V. The time constant of the measuring coil is equal about 15 ms (inductance $= 0.5$ H and resistance $\approx 34$ Q). Because this time is tens thousands times less in comparison to period of triangle wave, the shape of the current waveform is just as triangular as for the voltage waveform. Due to dynamic effects are imperceptible in current – time curve, the current value can be calculated according to Ohm’s law for direct current. The Eqs. (3) and (6) present current – time dependence in the supply coil. The AC bias (sinusoidal waveform) current $i_{AC}(t)$ (4) is the result of dividing the AC bias voltage component in Eqs. (1) and (2) by the supply coil impedance. Whereas the slow triangle wave component is divided by coil resistance only (5).

\[ i(t) = i_{AC}(t) + I_{r} \quad (3) \]
\[ i(t) = \frac{a}{\sqrt{R^2 + (aL)^2}} \sin(a(t + \phi) \quad (4) \]
\[ I_{r} = \frac{b}{R} \frac{n(t) - 2047}{2047} \quad (5) \]
\[ i(t) \approx \frac{a}{\sqrt{R^2 + (aL)^2}} \sin(a(t + \phi)) + \frac{b}{R} \frac{n(t) - 2047}{2047} \quad (6) \]

where: $I_{r}$ – current in the supply coil, $i_{AC}$ – sinusoidal (AC bias) current component, $I_{tr}$ – slow triangle wave current component (is considered as a constant value in short time), $L$ – supply coil inductance, $R$- supply coil resistance, $\phi$ – phase shift.

The inductance of the supply coil depends to some extent on the tested material. Additional experiments with the Hameg 8118 RLC impedance bridge have shown that the impedance module of supply coil is equal to 3194 Ω for empty (free air), 3547 Ω for FeSi (transformer steel) sample and 3314 Ω for low carbon steel sample. These values were measured under conditions: frequency of 1 kHz and voltage amplitude 1 V in the bridge – which is well below the saturation area. The changes in the impedance value of the supply coil are not significant but nevertheless influence to current AC bias amplitude in the supply coil and in consequence to AC bias voltage in the measurement coil. Hence, an auto-calibration procedure has been developed. Before the beginning of main measurements, the short auto-calibration procedure reads electrical parameters of coils (in a wide frequency range) in case with and without a sample. Next, the received AC bias signal is corrected in the microcontroller and in effect, discrepancies of the inductance in the supply coil for different samples are mitigated. The typical value of current in the supply coil covers the range of 40 µA–100 µA RMS for AC bias (dependently of frequency and other settings) and for slow triangle wave covers the range of -175 mA to +175 mA DC. The magnetic field strength produced by the supply coil is proportional to current in this coil (7). That in turn induces the appropriate current in the measurement coil. The electromagnetic force induced in measuring coil (8), (9), and (11) depends on change rate of magnetic flux (8) according to Faraday’s law.

\[ H(t) \sim i_{r}(t) \quad (7) \]
\[ u_{n}(t) = -k_{1} \frac{d\phi}{dt} = -k_{1} \frac{dH}{dt} \quad (8) \]
\[ u_{n}(t) = -k_{2} \mu \left(\frac{dI_{AC}}{dt} + \frac{dL}{dt}\right) \quad (9) \]
\[ |u_{AC}| = k_{1} \mu \frac{\cos(\omega t + a) \pm U_{DC}}{\sqrt{2}} \quad (10) \]
\[ |u_{AC}| \sim \mu(I_{r}) \quad (12) \]

where: $\phi$ - magnetic flux inside the measuring coil, $\mu$ – incremental magnetic permeability of a sample, $U_{DC}$ – voltage on terminals in the measuring coil, $|u_{AC}|$ – RMS value of AC bias voltage on terminals in the measuring coil, $k_0$, $k_1$, $k_2$ – equations factors which include i.a. geometrical dimensions of measurement coil, number of turns and sample to coil cross section area ratio.

The voltage induced in the measuring coil (10) contains AC bias (cosinusoidal expression) component and a direct current voltage component ($U_{DC}$). The sign (+ or -) of $U_{DC}$ depends on actual triangle wave edge of current in the supply coil – is it positive going or negative going. It should be noted, the value of $U_{DC}$ voltage is much lower than induced AC bias signal. This follows from the Eq. (9), where $dI_{AC}/dt$ expression is tens or hundreds times higher in comparison to $dI_{tr}/dt$. A typical $U_{AC}$ voltage level in the measuring coil is significantly less than 1 mV. Tests carried out with Rohde & Schwarz RT82002 oscilloscope and Keithley 2000 multimeter have confirmed the above considerations. The input amplifier and filters in the electronics unit (Figure 3) can easily separate and eliminate $U_{DC}$ in the signal received from the measuring coil. A typical level of the induced AC bias voltage in the measuring coil at frequency 400 Hz is about 15 mV pp (peak to peak) for condition without sample (this voltage is independent of the operating point – independent of $I_{r}$ level) and is about 90 mV pp for FeSi sample when the operating point has coordinates $H = 0$; $B = 0$. Finally, an analysis of Eq. (9), (10), and (11) leads to the conclusion, the induced AC bias voltage is proportional to the incremental magnetic permeability of a sample (12) in any operating point in the range of $-H_{max}$ to $+H_{max}$. One full measurement cycle relies on measuring $|u_{AC}|$ for each operating point, which changes according to Eqs. (1) and (5). Next, the $|u_{AC}|$ voltage vs. $I_{r}$ current dependence is numerically integrated and in effect the magnetic hysteresis loop of the tested material is obtained. It should be note, if the AC signal were too strong in the supply coil, then it may cause a relatively large reentrant loops on the main loop, an electrical signal distortion in the measuring coil and consequently an inability to the correct determination of the main loop hysteresis. To avoid excessive distortion the AC bias amplitude should be as low as possible. But, on the other hand, if the amplitude of the AC bias in the supply coil is a very low, then the AC bias voltage induced in the measuring coil is a very low too and is very close to a noise level – sometimes it could be too close to this level or even in it. Consequently, SNR (signal to noise ratio) could be too low to correct determine the hysteresis loop. In the presented system, the
SNR level is equal about 600–700 (in a linear scale V/V) for the amplified signal from the measuring coil. The measurement uncertainty of the system is approximately equal to: for magnetic flux density 0.04% ± 5.0 mT and for magnetic field strength 0.13% ± 1.5 A/m. All these values at 21 °C were obtained. All measurements were started 10 min after switching power on the device.

The presented set-up offers magnetic field up to +/- 3 kA/m. Due to the diameter of a wire in the supply coil is relatively thick (1 mm), the current could be easily increase even 10 times (up to level 1.75 A) with no risk of overheating – the power dissipation (heat) would be equal about 104 W and that is not too much for coil weighing over of 10 kg and outside surface area over 1500 cm². In consequence, it is possible to obtain the magnetic field in order of +/- 30 kA/m. Of course it requires to construct a new “stronger” power amplifier (Figure 3) in the electronics unit.

3. Results and discussion

The results presented below describe the study of two reference materials: FeSi transformer steel (soft magnetic), and low carbon steel (material with higher coercivity in comparison to the first sample). Both samples have the same dimensions (0.4 × 10 × 200 mm). These materials with well-known properties were deliberately chosen for the study, because they give the opportunity to evaluate the correctness of the new just presented method. All measurements were carried out in temperature about 295 K. At the initial researches, it was checked whether the tested material was getting hot as a result of e.g. eddy current formation or heat transfer from the supply coil. The true H values have been determined by taking into account dimensions of the supply coil, its number of turns and number of layers. Maximal magnetic field strength produced by the presented system is up to 3 kA/m in the central coils point. The estimation of the true values of the magnetic flux density B is a bit more complicated. It follows from sample to coil cross section area ratio and first of all the demagnetizing field effect expressed by the geometry dependent demagnetizing factor [5, 6]. In this effect, the resultant permeability of sample could be hundreds or thousands times less than true permeability of the tested material and value of sample's permeability depends on local value of H. Therefore, for simplicity, the values of B were estimated approximately using the reference FeSi sample. Due to the aim of researches are not quantitative measurements, this solution has accepted. The characteristics of that reference sample were determined formerly using Fok-Gyem hysteresosgraph (certificated apparatus, made by Fok-Gyem). Based on these characteristics, the presented system was initially calibrated. At the moment, a high precision calibration is not the most important. Currently ongoing works are focused on even better adaptation of the presented method for testing the properties of nanomaterials. Results of the magnetic investigation in nanomaterial samples will shown in another paper.

The presented equipment has implemented auto-calibration function and it runs every time at the beginning of measurement series (each series of measurements consists of one or more cycles). This function detects e.g. a reference background level (for empty measuring coil – without any sample), maximum input voltage for a particular sample and it demagnetizes the sample. During the measurement process, for any single measurement point, the received voltage from the measuring coil is subtracted from the voltage of background (determined earlier while calibration). The following results were obtained for a period of the triangle wave signal set at 360 s and AC bias frequency was set at 400 Hz. The Figure 4 shows magnetic hysteresis loop for the FeSi sample.

The first cycle is evidently shifted to the left up and it is not symmetrical relative to the origin point. However, while each subsequent cycle, the hysteresis shift successively towards to the to right down. The hysteresis loop becomes symmetrical to the origin point typically after about 20–30 cycles. After these number of cycles, the shifts getting smaller. Because the starting point is not visible in Figure 4, the middle sector of Figure 4 was zoomed in Figure 5.

The Figure 6 shows hysteresis loops for the low carbon steel sample. As in the previous case, the first cycle of the hysteresis loop is shifted and next the loop shifts (while next cycles) towards to the fourth quarter. The surface area of the hysteresis loop in the low carbon steel is clearly many times bigger in comparison to the FeSi loop in Figure 4.

To check whether the hysteresis loop shifts (observed in Figures 4, 5, and 6) are (or are not) the result of numerical integration errors and the result of parameter drift in the electronics components, in-depth investigation of a drift of an operating point in the system has been performed. The drift of the operating point in circuits is a well-known problem in the field of electronics engineering. This is caused most frequently by temperature changes in electronics components in a circuit. Due to the hysteresis loop shift is observed, the drift of the operating point in electronic systems and numerical algorithms in the program code have been carefully investigated. The Figure 7 presents B(H) dependence for 30 cycles for the module without the sample (while the measuring coil is empty). The curves for all cycles have overlapped one another and in effect a single line in the below plot is seen.

Figure 4. Hysteresis loops of the FeSi sample for 1st cycle and last one 30th cycle.
For each cycle (for each loop – it means for a pair of curves) in Figure 7 which are seen as the lines (linear functions), the zero points were determined. Taking these zero points, Figure 8 was created. The zero points locations during increasing and decreasing of the magnetic field strength are shown in the Figure 8. The values of offsets of the zero points (their drift in next cycles) are low and they are typical a few A/m. Many measurement series have indicated a random character of these low drifts of the zero points. The quantized character of levels of the points in Figure 8 is a result of a finite resolution of DAC and ADC in the electronics unit.

Carried out measurements have indicated that the hysteresis loop shift in Figures 5 and 6 is not a result of a drift of an operating point of electronics system and it is not a result of successive growing of error while numerical integration process. All measurements were always started after a minimum of 10 min from turning on the power. The hysteresis loop shift effect is related to the method, material physical properties and it forces the position of loop in the H–B coordinate system while the initial cycles. The hysteresis shift effect is not widely known in literature. Shifted hysteresis loop is observed in thin-film heterostructures [7] and it is related to an effect of exchange bias on the hysteresis loop of a ferromagnetic layer coupled to an antiferromagnetic layer. It should be note, in that last case the loop position (in the B(H) coordinate system) is steady for unchanged measurement conditions and there is not a drift of the loop during the next cycles. So, that above example of hysteresis loop shift cannot be compared to results in the presented paper. The hysteresis loop shift effect and its moving to steady localization is briefly described in book [11]. Author indicates that the exact symmetry of the hysteresis curve relative to the origin of the B–H coordinate system is obtained only after several periodic loops, during which one gradually enters the rhythm of the final curve. The proposed method allows to conduct investigations for various AC bias frequency. The Figures 9 and 10 show specific for each sample the spectral

Figure 5. Middle sector of Figure 4, zoomed in the range of -200 to 200 A/m.

Figure 6. Hysteresis loops of the low carbon steel sample for 1st cycle and last 30th cycle.
characteristics of $|u_{\text{mAC}}|/|u_{\text{ref}}|$ ratio (AC bias voltage to reference voltage ratio) for static magnetic field strength of 0 A/m (starting point) and 3 kA/m (saturation region). The reference voltage $u_{\text{ref}}$ is the induced voltage in the empty measuring coil (without sample). A spectral characteristic of $u_{\text{ref}}$ is always measured and is written to microcontroller memory during the calibration process. This characteristic is varying about $\pm 15\%$ in function of frequency in the range of 10 Hz - 1 kHz. The maximal amplitude of the $u_{\text{ref}}$ is received at 180 Hz. Only by knowing the $u_{\text{ref}}$ spectra it is possible to determine the true spectral characteristic for any investigated sample. The $|u_{\text{mAC}}|/|u_{\text{ref}}|$ ratio at individual frequency reflects a value of relative permeability at this frequency.

The characteristic in Figure 9 shows clearly that a magnetic features of the FeSi sample (mainly a permeability) are sensitive for frequency changes. Increasing the frequency reduces the $u_{\text{mAC}}/u_{\text{ref}}$ ratio. The level of measuring points for the low carbon steel sample (in Figure 9) is almost unchanged and this fact distinguishes that steel from the FeSi. The spectral characteristic in a saturation region – Figure 10 (magnetic field strength of 3 kA/m) looks completely different in comparison to the previous case. There are little differences in results between samples in Figure 10. The measuring points for low carbon steel are situated minimal above points for FeSi and they change to a small extent around level of 1.31. The level of the points for FeSi sample is minimal lower. It should be noted that the slight fluctuations in value in Figure 10 fall within the scope of measurement uncertainty of the presented equipment. The frequency influences to hysteresis loop shape in a similar way, but not exactly the same like in the case of standard methods. An increase in Figure 7. Magnetic flux density vs. field strength inside the measurement module for setup without investigated sample during 30 measurement cycles (the measurement points for each cycle have overlapped one another and in effect a single line in the plot is seen).

Figure 8. Exemplary offsets of zero points for curves in Figure 7 while: ▪ increasing the magnetic field strength from $-H_{\text{max}}$ to $+H_{\text{max}}$, ● decreasing from $+H_{\text{max}}$ to $-H_{\text{max}}$. 
frequency causes a slight increase in the width of the hysteresis loop and simultaneously a significant decrease in the saturation flux density level.

4. Conclusions

The results of the study show a comparative research method for magnetic properties investigation, which in technical physics and material science (magnetic alloys) could be applied. An important advantage of the presented method is the opportunity to stop changes of the magnetic field at any operating point in the B(H) characteristic. Next, it is possible to deeply investigate local properties of a sample, using AC bias field for various frequencies. The frequency dependence of the magnetic hysteresis gives important information about the properties of the tested material. It is also the subject of theoretical considerations in paper [8, 9]. The presented method is based on mixing two magnetic fields in the tested material: the first of these fields has a very low amplitude, the shape of sine wave (AC bias) and the frequency range form several to thousands of Hertz. The second field has a very low frequency (order $10^{-2}$ Hz or less), the high amplitude up to $\pm 3$ kA/m and approximately a form of the triangle wave. This slow triangle wave field could be considered as a static field, which determines the operation points in the B(H) curve. Thus, the new solution has some features of both: static and dynamic methods of the hysteresis testing. Moreover, carried out numerous investigations have revealed the effect of shifting of hysteresis loop while the initial 20–30 cycles. Using the experience gained, it is planned to develop the next generation of the presented device, which will give higher precision and even lower voltage of AC bias. The application of the measuring coil (use of the Faraday’s law of induction) allows detecting AC bias signal with the best accuracy and ranges for low magnetic field in comparison to other methods [10]. To measure precisely magnetic parameters of any material, it is needed the calibration with reference samples. Ultimately, the measurement procedure will
assume that for each investigated sample, two reference samples are prepared. All samples (examined and references) in that procedure will have the same dimensions. Reference samples are made of materials with well-known properties (hysteresis loop widths etc.) and described in literature (tested on various hysteresographs). Next, measurements results for the tested sample and reference samples are compared and in effect the hysteresis loop in the tested sample is determined (calculated). Of course, it is advisable to use more than two reference samples. The above procedure simplifies the consideration and the post-measurement calculations of the demagnetization effects in the tested material. These calculations are realized after measurements and sending data from the device to a computer (any computer with USB port). The FeSi steel and the low carbon steel are very well known materials and they are good candidates to reference materials. Their characteristics are written to microcontroller memory. Of course, for the highest accuracy, individual calibration is also available before each series of measurements. This is not a disadvantage because the most of instrumental analysis methods require reference samples (materials). The presented set-up offers magnetic field in the range of +/- 3 kA/m, but the supply coil design (dimensions, weight) allows to increase current even up to 10 times and then the magnetic field would increase up to +/- 30 kA/m. This gives the opportunity to study a wide variety of materials.

Declarations

Author contribution statement

A. Nowrot: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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The authors declare no conflict of interest.

Additional information

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