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Continuous-wave far-infrared ESR spectrometer for high-pressure measurements

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ABSTRACT

We present a newly-developed microwave probe for performing sensitive high-field/multi-frequency electron spin resonance (ESR) measurements under high hydrostatic pressures. The system consists of a BeCu-made pressure-resistant vessel, which accommodates the investigated sample and a diamond microwave coupling window. The probe's interior is completely filled with a pressure-transmitting fluid. The setup operates in reflection mode and can easily be assembled with a standard oversized microwave circuitry. The probe-head withstands hydrostatic pressures up to 1.6 GPa and interfaces with our home-built quasi-optical high-field ESR facility, operating in a millimeter/submillimeter frequency range of 105–420 GHz and in magnetic fields up to 16 T. The overall performance of the probe was tested, while studying the pressure-induced changes in the spin-relaxation mechanisms of a quasi-1D conducting polymer, KC_{60}. The preliminary measurements revealed that the probe yields similar signal-to-noise ratio to that of commercially available low-frequency ESR spectrometers. Moreover, by observing the conduction electron spin resonance (CESR) linewidth broadening for KC_{60} in an unprecedented microwave frequency range of 210–420 GHz and in the pressure range of up to 1.6 GPa, we demonstrate that a combination of high-pressure ESR probe and high-field/multi-frequency spectrometer allows us to measure the spin relaxation rates in conducting spin systems, like the quasi-1D conductor, KC_{60}.

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1. Introduction

The high sensitivity and resolution of electron spin resonance (ESR) makes it a particularly valuable technique for elucidating details of the nature and local environment of electronic spins. Commercially available and home-built ESR systems usually operate at low microwave frequencies ranging from 3 GHz (S band) to 35 GHz (Q band). Among them, the most popular ESR spectrometers operate at ~9 GHz (X band). The highest-frequency ESR spectrometer now commercially available works at ~90 GHz. Recently, however, an increasing number of research laboratories have become interested in systems that operate at higher microwave frequencies (90–750 GHz) [1,2–4]. The rationale for extending the microwave radiation frequency range beyond the traditional lower microwave bands was to provide improved spectral resolution and higher overall sensitivity at corresponding higher magnetic fields. The high-field technology makes it also applicable to non-Kramers spin systems, i.e. integer spin systems, having large zero-field splitting. Such a spin systems are often ESR-silent at conventional low microwave frequencies and low magnetic fields. Moreover, the high-field/high-frequency approach opens up new avenues for multi-frequency scanning experiments. Such methodology is extremely valuable for studying numerous spin systems that otherwise would be difficult to investigate by traditional low-field/low-frequency means [5–7].

In the great majority of low- and high-frequency ESR experiments reported so far, besides of varying usual instrumental setting, such as microwave power or magnetic field modulation amplitude and frequency, the experimental variables have been limited to the sample temperature as the only variable physical parameter of the system under study. It is widely accepted, however, that hydrostatic pressure is a powerful tool for altering the physical properties of condensed matter. In particular, with the use of hydrostatic pressure, thermodynamic, magnetic and electronic properties of materials can be varied in an elegant and controllable manner (for a recent review, see, e.g. [8, and references therein]). For the above-mentioned reasons, hydrostatic pressure has been used historically as a control parameter for studying phase transitions by electrical transport measurements, luminescence, nuclear magnetic resonance (NMR), Mössbauer and Raman spectroscopies, as well as X-ray and neutron diffractions. ESR is particularly suitable for detecting phase transitions (e.g. metal–insulator transitions, changes in magnetic order, polymerization, etc.), hence several laboratories tried to explore a possibility of performing ESR experiments under hydrostatic pressure [9–17]. However, despite its obvious potential, a combination of ESR with hydrostatic pressure has not become widely popular.

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A major difficulty in designing hydrostatic high pressure ESR systems arises from topological and technical constraints related to the coupling of high-frequency electromagnetic radiation to the microwave resonant structures. Commercially available low-field ESR spectrometers, operating at atmospheric pressure, are usually equipped with metallic-walled cavities whose dimensions are comparable to the microwave free-space wavelength (e.g. ~3.5 cm at 9 GHz). A major motivation for using cavities is to increase the intensity of the magnetic component of the incident electromagnetic radiation, B_1, at the sample position, which leads to a higher overall sensitivity of the setup. Although technologically feasible, the design of such voluminous resonant structures (~50 cm^3), capable of withstanding high hydrostatic pressures, would result in rather unpractical devices. For example, the external dimensions of corresponding pressure-resistant vessels would be too large to be accommodated in stardard fixed-gap laboratory electromagnets. Beyond the criterion for withstanding pressure-generated mechanical strain, there are also other requirements for high-pressure ESR probes. Clearly, the bulky walls of the pressure-resistant vessels must be strictly non-magnetic. Moreover, the customarily used signal detection scheme requires magnetic field modulation at the sample space. The skin depth for the most widely used 100 kHz magnetic field modulation is of the order of 0.2 mm, therefore low-frequency magnetic field modulation has to be used (<1 kHz), which is capable of penetrating thick metallic walls of the pressure-resistant vessels [11,18]. Although an alternative solution, namely the insertion of the field modulation coil inside the pressurized volume, is technically conceivable [19], it complicates the design by the necessity of making pressure-resistant electrical feed-throughs.

The approaches presented in the literature approaches for combining high hydrostatic pressure with ESR involve two major routes. One of them uses cylindrical cavities entirely filled with low-loss high-dielectric materials in order to reduce the active volume, leading to smaller external dimensions of the ESR probes [10,11,18,19]. Usually, such microwave cavities are inserted directly into the pressure-resistant vessels made of beryllium copper (BeCu), a non-magnetic alloy with excellent mechanical properties. The high dielectric filling provides a reliable pressure-sealed microwave coupling to waveguides [18,19], as well as reducing the cavity size, which scales down as $1/\sqrt{\varepsilon_r}$, where ε_r denotes the relative dielectric permittivity. The other design approach involves a combination of gasketed diamond- or sapphire-anvil cells with specialized microwave resonanting structures, such as a plate resonator [14] or a dielectric resonator [17]. In both cases, the pressure-generating anvil cells are surrounded by miniature microwave resonant structures that confine the microwave component, B_1, to the pressurized volume, for example the sample hole of the metallic gasket.

To date, a combination of high-field/high-frequency ESR system with a high hydrostatic pressure vessel operating above 1 GPa has been reported by only one group [20]. It is a pulsed high-field, multi-frequency (60–700 GHz) system, that operates up to 17 T and covers the hydrostatic pressure range up to 1.1 GPa. It features a non-resonant transmission-type pressure vessel, which is equipped with two microwave-transparent sapphire plugs. This pressure vessel is located in the same cryostat compartment as the InSb microwave detector, which limits the operating temperature range to 1.8–4.2 K. Notwithstanding the above technical limitation, the non-resonant high-frequency approach described in [20] offers a remarkable simplification of the overall system design, because the complicated resonant microwave structures can be avoided without sacrificing the spectrometer performance [2,21].

Here, we report on a newly designed high-field/high-frequency ESR probe that operates up to 1.6 GPa and in the temperature range 1.5–300 K. The probe is connected to a quasi-optical high-field ESR spectrometer, operating in a millimeter/submillimeter frequency range of 105–420 GHz and in magnetic fields up to 16 T [22]. The design for the ESR probe is simple and robust. It is based on a non-resonant cylindrical pressure-cell that accommodates at its bottom the investigated sample and operates in reflection mode. The pressure-cell can easily be coupled to an oversized corrugated waveguide, which routes the microwave radiation from the microwave source to the sample and back to the detector. The external diameter of the pressure-cell is 25 mm, which make it compatible with the inner bore of a commercial continuous-flow variable temperature insert (VTI) from Oxford Instruments with sample access diameter of 30 mm. A plug with a Poulter-type microwave-transparent diamond window provides coupling to the waveguide, as well as serving as a pressure seal. The pressure-cell is entirely filled with a pressure-transmitting fluid: Daphne oil 7373. The microwave coupling window is transparent to visible light, therefore the hydrostatic pressure can be easily measured using a standard ruby-fluorescence technique. A low-frequency (300 Hz) magnetic field modulation is provided by an external coil. It is worth noting, however, that the pressure-cell can also operate using our newly-developed microwave frequency-modulation scheme [22]. This latter strategy, based on modulating the frequency of the incident microwave radiation, rather than applying a conventional magnetic field modulation, has been found very sensitive and useful for detecting ESR signals from weak paramagnetic samples.

The overall performance of the ESR probe was tested while following spin-relaxation changes in a quasi-1D conducting polymer, KC_{60}. The setup allowed us to observe the pressure-induced evolution of the CESR linewidth in polycrystalline KC_{60} up to 1.6 GPa and in an unprecedented microwave frequency range of 210–420 GHz.

2. Experimental

The overall scheme of the high-frequency/high-hydrostatic pressure ESR spectrometer is depicted in Fig. 1. The spectrometer operates in reflection mode in a Faraday configuration, with microwave propagation vector of the incident radiation aligned parallel to the external magnetic field. 13.1 GHz microwave radiation is

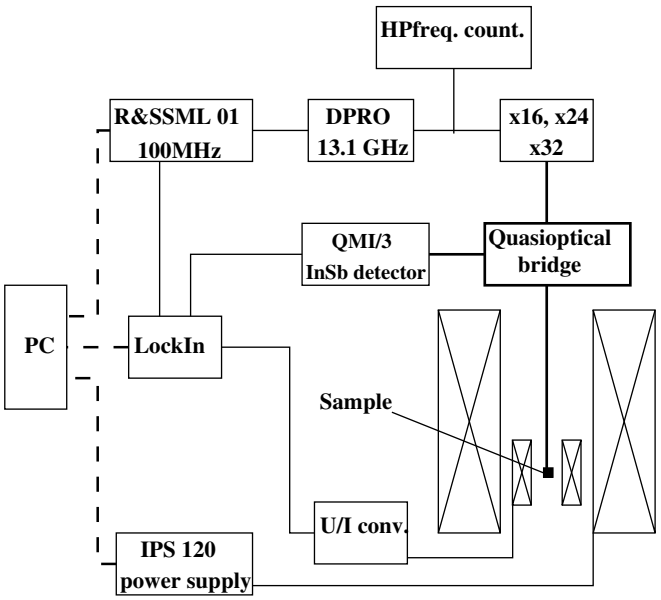

Fig. 1. Block diagram of the 105–420 GHz continuous-wave ESR spectrometer working in reflection mode, utilizing either frequency or field modulation. Dashed lines mean the GPIB communication. More detail is given in the text.

generated by a phase-locked dielectric resonator oscillator, Herley-CTI DPRO, which is locked to a 100 MHz Rohde&Schwarz, Model SML01 signal generator. The 13.1 GHz base frequency is upconverted by a chain of frequency doublers and triplers (Virginia Diodes) to reach the 105–420 GHz sub milimeter frequency range with 10^{-9} relative frequency stability. The available microwave power is ~2 mW at 210 GHz. The light is guided to the sample through a quasi-optical bridge (Thomas Keating Instruments) and back to a liquid helium cooled InSb hot-electron bolometer (QMC Instruments).

The pressure-cell, with the investigated sample inside, is loaded into the VTI and positioned in the center of the superconducting magnet. The VTI operates over the temperature range 1.5–300 K, using the dynamic flow of helium gas or liquid through a heat exchanger below the sample space. The overall temperature accuracy of the system is of 0.1 K.

As can be seen from the block diagram shown in Fig. 1, the high-frequency/high-hydrostatic pressure ESR spectrometer can operate using two schemes for signal detection: (i) conventional field-modulation and (ii) microwave frequency-modulation [22].

For acquiring a conventional cw-ESR spectrum with magnetic field modulation, the low-frequency (300 Hz) signal is provided by the internal oscillator of a lock-in amplifier, Stanford Research Systems, Model SR830, which is also used for phase-sensitive signal detection. Before energizing the modulation coil, the modulation signal is fed into a home-made voltage-controlled current source (U/I converter). The output current of the U/I converter is linear up to a maximum current 0.5 A and a maximum frequency 20 kHz. The current source is needed instead of the voltage source of the lock-in to have a temperature-independent magnetic field modulation amplitude.

The modulation coil is wound directly on the pressure-cell body and delivers effective peak-to-peak amplitude of up to ~0.1 G at the sample position.

For performing ESR measurements with the newly-developed scheme of signal detection, based on microwave frequency modulation [22], we modulate the 100 MHz reference signal of the dielectric resonator oscillator. The frequency- and phase-noise of the millimeter-wave is determined by the noise of the Rohde&Schwarz reference oscillator and has been found to be much lower than the noise originating from the mechanical vibrations of the spectrometer. During the phase-sensitive detection of the ESR signal with modulation of the microwave frequency the reference signal was provided by the Rohde&Schwarz signal generator.

The high-frequency/high-pressure probe operates using either the standard magnetic field-modulation or employing our newly-introduced technique of modulation of the carrier microwave frequency [22]. It has been demonstrated that for low frequency modulation depths (up to 10 MHz) both modulation techniques yield fully equivalent line shapes.

In all the experiments reported herein, independently of the modulation scheme used, conventional field-swept ESR traces were acquired. As can be seen in Fig. 1, the quasi-static magnetic field is provided by an Oxford Instruments superconducting magnet, with a maximum field of 16 T and a field homogeneity of 10^{-5} in 1 cm^3. Data acquisition is performed via a GPIB interface connected to a PC. Triggering of the magnetic field and data acquisition is synchronized by a signal from a function generator (Agilent 33250A).

Fig. 2 shows a simplified cross-sectional view of the pressure-cell. The pressure-resistant vessel is made of BeCu and is cylindrical in shape. The outer diameter is 25 mm and fits conveniently into our continuous-flow cryostat. The overall design is very similar to our previous pressure-cell that was manufactured for infrared spectroscopy [23].

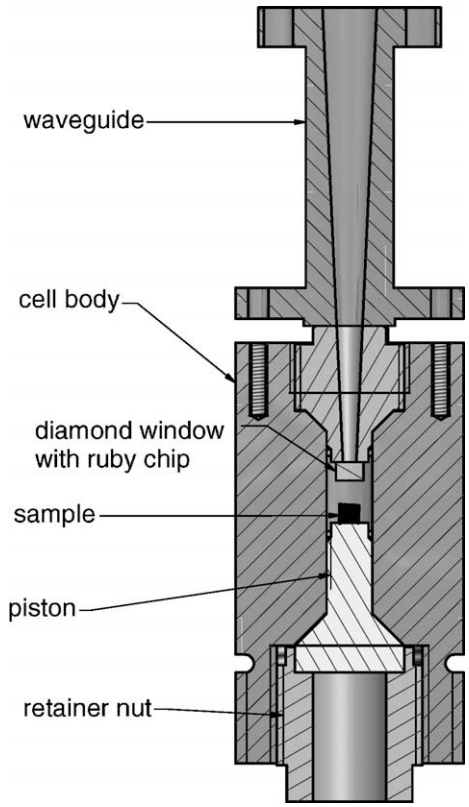

Fig. 2. Simplified cross-section of the clamped-type piston cylinder pressure cell for high-frequency ESR measurements.

The microwave radiation enters the pressurized volume through a Poulter-type diamond window. The opening of the window is 1.5 mm in diameter, which is sufficiently large to allow measurements at and above 210 GHz. The filling of the most narrow part of the conical section of the waveguide with a low-loss dielectric material, like polytetrafluoroethylene (PTFE), allows operation also at 105 GHz. The inner diameter of the pressure-cell is 5 mm and the average length of pressurized volume is ~15 mm, the inner volume of the pressure-cell can be considered as a largely oversized cavity, even at the lowest microwave frequency.

The top flat surface of the piston serves as a bottom mirror for the incident microwaves. The investigated sample is placed as close as possible to this bottom mirror surface, because at this location the magnetic component of the microwave field is at its maximum for any frequency. Daphne oil 7373 is used as a pressure-transmitting fluid, since its properties have been well characterized [24–26]. We also found Daphne oil well suited for studying numerous materials, including organic crystals, which represent the major subject of interest in our group. The standard ruby-fluorescence technique is employed to measure the hydrostatic pressure in the pressurized volume inside the BeCu body. The intense fluorescence of the ruby's R-lines (Cr^{3+}-related luminescence in Al_2O_3 with Cr^{3+} impurities) is sensitive to the change of the interatomic distances, when an external pressure is applied. In the experimental setup presented in this work, the pressure-induced shift of the R-lines was monitored using a commercially available Raman spectrometer Dilor, Model SP300 (Dilor SA, Lille, France). This system consists of an argon laser and a multichannel spectrophotometer, equipped with a photodiode array detector. The laser beam (30 μm in diameter) from the Dilor's argon laser is routed to a small ruby crystal glued directly to the diamond window. The green laser light, $\lambda = 514.5$ nm, excites the transitions within the ruby Cr^{3+} electronic manifold, which gives rise to the red fluores-

cence around $\lambda = 694$ nm. As can be seen in Fig. 2, the BeCu window-support has a conical opening, which makes it possible to photo-excite the ruby crystal in the sample compression chamber as well as facilitating the routing of the red ruby luminescence back to the Dilor's multichannel photodiode array detector.

3. Results and discussion

The overall performance of the setup was checked by acquiring the CESR signal from a quasi-1D conducting polymer, KC_{60}.

This compound, an orthorhombic phase conducting polymer, is a representative of a class of fullerene-based alkali-doped materials, AC_{60}, where A = K, Rb, Cs. From the experimental standpoint, the polycrystalline KC_{60} features a strong, nearly Lorentzian in shape CESR signal, which can readily be observed at room temperature. When measured at low microwave frequency of 9 GHz, at room temperature and ambient pressure, the fine powder of KC_{60} yields a strong and relatively narrow CESR signal ($\Delta H \sim 0.45$ mT) [17]. For these reasons, KC_{60} has already been used for calibration of other ESR systems [17,27]. Previous studies of the magnetic susceptibility and electrical transport properties in a wide temperature range (50–350 K) confirmed the metallic-type behavior of the orthorhombic phase KC_{60} [28]. In particular, the ESR-measured magnetic susceptibility of KC_{60} was found to be a temperature-independent Pauli-like susceptibility. Moreover, the ESR peak-to-peak line width measured over this temperature range revealed only a weak linear dependence, which can be understood in the framework of the Elliott model of electronic spin relaxation through combined effects of resistivity scattering (electron–phonon interactions) and spin–orbit interactions [28]. For the orthorhombic phase of KC_{60} this type of behavior results from a quasi-1D electronic structure of the polymerized C_{60} chains. Such polymer chain structure accounts also for a strong spin susceptibility as well as non-conventional pressure dependence of spin relaxation and ESR linewidth in KC_{60} [29,30]. Thus, due to almost negligible temperature dependence of both ESR susceptibility and linewidth, all the test measurements reported herein were performed at room temperature.

Before assembling the pressure-cell, a small amount of polycrystalline KC_{60} was transferred into a 0.4 mm inner-diameter polyethylene tube and positioned at the top surface of the piston. The instrumental settings were as follows: field-modulation frequency 300 Hz, 2 mW microwave power, 30 ms time constant, 1000 points per scan, 30 s per scan and 0.1 G magnetic field modulation amplitude. While acquiring ESR traces with microwave frequency modulation, we used 10 kHz modulation frequency with 4 kHz modulation depth.

To estimate the sensitivity of the setup, the same KC_{60} polycrystalline sample was also measured using a conventional ESR spectrometer operating at 9 GHz, Bruker ESP 300 E. These comparative measurements revealed that our newly-developed high-pressure/high-frequency ESR setup, which operates in reflection mode without a well tuned single mode microwave cavity, yields similar sensitivity to that of commercially available X-band ESR spectrometers. For each pressure setting, the ESR traces were acquired at three microwave frequencies of 210, 315, and 420 GHz. These frequencies were obtained by frequency multiplication, using a chain of frequency doublers and triplers (Virginia Diodes). Changing the microwave frequency was achieved without any major modification to the spectrometer configuration, without moving the sample in particular, this is important in the case of anysotropy measurements on single-crystals.

Prior to ESR measurements, the hydrostatic pressure was increased in steps of 0.2 GPa controlled by the load on the hydraulic press. Subsequently, the precise hydrostatic pressure inside the pressure-cell was measured with the ruby-fluorescence method.

The hydrostatic pressure-induced evolution of the CESR linewidth observed at 210, 315 and 420 GHz at room temperature for a polycrystalline KC_{60} is shown in Fig. 3. The CESR linewidths were determined by fitting the experimental spectra to the first derivative of a Lorentzian function. The observed linear increase of the CESR linewidth with increasing hydrostatic pressure shown in Fig. 3 is similar to that reported in [17] and points to a spin-relaxation mechanism that is governed by the inter-chain spin-flop coupling between the conducting chains, thus confirming the hypothesis of the existence of polymeric chains and quasi-1D-conducting character of KC_{60} [29]. Remarkably, the plot of the pressure-induced evolution of the CESR linewidth in KC_{60} (inset to Fig. 3) points to unusual quadratic frequency dependence. Similar frequency-related perturbation of the electronic spin relaxation and de-phasing, resulting in CESR linewidth broadening, has been observed and reported for many conducting spin systems [31].

Although it can be explained in terms of changes in the spin dynamics, induced by increasing magnetic field and microwave frequency, the more quantitative evaluation of this mechanism is beyond the scope of the present work. Clearly, however, the above findings demonstrate that the ESR high-frequency/high-pressure probe presented herein, operating at millimeter and submillimeter microwave frequencies, is capable of detecting the time scale of the spin dynamics in systems containing spins of conduction electrons, like in the quasi-1D conductor, KC_{60}.

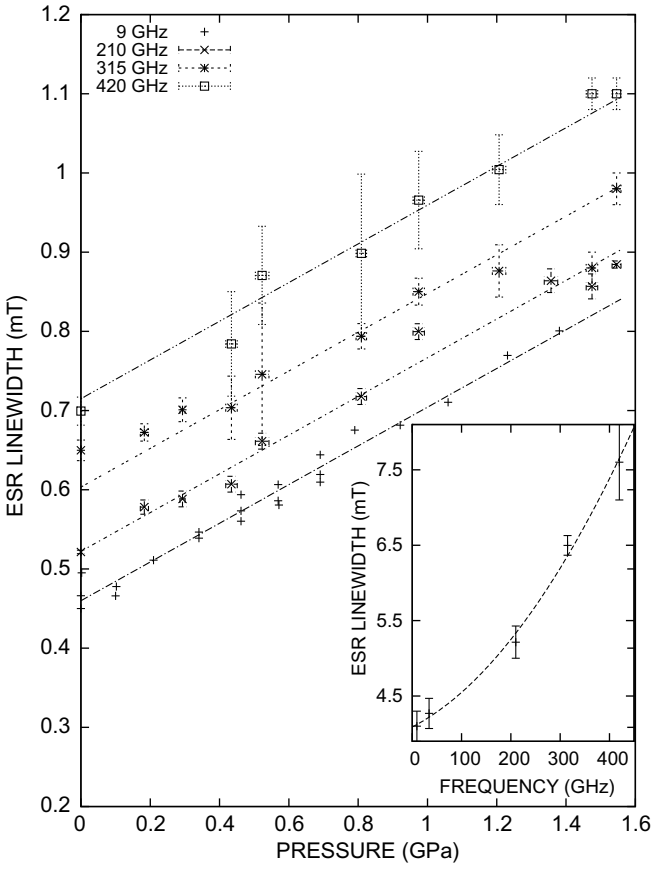

Fig. 3. CESR linewidth of a polycrystalline KC_{60} sample as a function of hydrostatic pressure at room temperature. The measurement was done at three different microwave frequencies 210, 315 and 420 GHz. The 9 GHz data were taken from Ref. [17]. Inset: evolution of the CESR linewidth as a function of microwave frequency observed at room temperature at ambient pressure.

The linear pressure dependence of the CESR linewidth shown in Fig. 3 points to a possibility of using KC_{60} as an internal pressure gauge for monitoring pressure in situ.

4. Conclusions

We have presented a newly-developed setup for performing high-field ESR experiments under high hydrostatic-pressure in a broad range of millimeter and submillimeter microwave frequencies. The device is simple and robust and can easily be coupled to the high-frequency quasi-optical ESR spectrometer operating in our laboratory. The design represents a substantial improvement in both accessible pressure range and sensitivity. Further improvement in the sensitivity is possible by placing a modulation coil inside the pressure-cell. We also demonstrate the feasibility of in situ pressure calibration by monitoring the pressure-induced broadening of the CESR feature of KC_{60}.

Acknowledgments

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