

多孔硅上银纳米粒薄膜的制备及其表面增强红外光谱

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摘要: 通过简便的化学沉积法在多孔硅上制备银纳米粒薄膜用于表面增强红外光谱检测。通过 Ag^+ 与多孔硅表面的 SiH_x 发生氧化还原反应将银纳米粒子沉积在多孔硅表面。红外探针分子溶解于无水乙醇中进而被均匀分散在多孔硅表面, 实验结果显示: 对氨基苯硫酚、对氨基苯甲酸和对氟苯硫酚 3 个探针分子的红外峰分别最大增强了 10、85 和 21 倍。银纳米粒的大小和形状等物理特性、探针分子是否有与银表面进行强结合的基团和芳烃结构、以及表面选律等因素影响表面增强红外的吸收效应。

关键词: 多孔硅; 银纳米粒; 表面增强红外光谱; SEIRA

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Facile Fabrication of Silver Nanoparticulate Films on Porous Silicon for Surface-Enhanced Infrared Absorption Spectroscopy

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Abstract: A facile chemical deposition method was developed to fabricate silver nanoparticulate films on porous silicon for surface-enhanced infrared absorption (SEIRA) spectroscopy. The silver nanoparticulate films were deposited on freshly anodized porous silicon through a spontaneous redox reaction between Ag^+ ions and surface SiH_x species. Probe molecules dissolved in ethanol were homogeneously spread on the porous membrane area with clear boundaries. The results show that three probe molecules of p-aminothiophenol, p-nitrobenzoic acid, and p-fluorothiophenol are enhanced on silver nanoparticle deposited porous silicon as 10, 85 and 21 times high as on bare porous silicon respectively. Several factors such as the particle size and shape, the probe molecular binding groups and aromatic structures, and surface selection rules are considered for contribution to the SEIRA effect.

Key words: porous silicon; silver nanoparticles; surface-enhanced infrared absorption spectroscopy; SEIRA

0 Introduction

Surface-enhanced infrared absorption (SEIRA) spectroscopy discovered by Hartstein^[1] has received great attention since 1980s. Owing to its high sensitivity, SEIRA has become one of the most useful analytical methods and has been used widely in

chemical, environmental and biomedical fields^[2-3]. SEIRA has been mainly observed on island films of the coinage metals such as Ag, Au, Sn and Cu. Among these metals, Ag is the most universal one because of its broad plasma resonance in the infrared spectral region and its high enhancement factor^[4]. There are two general strategies to fabricate a film of Ag nanoparticles

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(Ag-NPs) on a substrate surface: (1) Dry process, including vacuum evaporation, physical vapor deposition and sputtering^[5-6], is restive, expensive and time-consuming. (2) Wet process includes colloid-based coating, electrochemical galvanization and chemical deposition^[4,7-18]. Chemical deposition has received much attention because it is easy to realize in a general chemistry laboratory. Activation sites for infrared enhancement can be formed on some substrates by reducing metals on the pre-deposited seeds in the corresponding metal ion solution with a proper reducer. Several metal nanoparticulate films of Au, Ag, Sn, Cu, etc. have been fabricated by this method^[19-23]. The SEIRA effect is related to the size, shape, and aggregation of metal nanoparticles. It has been reported that Ag-NPs films consisted of closely crowded islands can result in strong surface enhancement^[24-25].

Many materials such as Si, Ge, CaF₂ and glass have been used as substrates^[26-32]. The surface properties of substrates also affect SEIRA because they decide the appearance of metal particulate films^[5]. Silicon is the most commonly used substrate for SEIRA measurements in the attenuated total reflection (ATR) mode^[2-3]. Since the ATR-SEIRA set-up is not easy and convenient to use, in this paper, we report a very simple method for SEIRA measurements by chemically depositing Ag-NPs on the porous metastable silicon hydride (SiH_x with $x=1, 2, 3$) surface. Due to the high specific surface area of porous silicon (PSi), the SEIRA spectra of probe molecules dropped on the Ag-NPs/PSi chip can be obtained easily and simply through the transmission mode. A probe molecule of *p*-aminothiophenol (*p*-ATP) was used to assay the appropriate deposition conditions for Ag-NPs films first. Then scanning electron microscopy (SEM) was applied to image the topographical structures of Ag-NPs films for structure-function association. Two other model probe molecules, *p*-nitrobenzoic acid and *p*-fluorothiophenol, also demonstrate excellent enhancement performance up to high magnification factors of 85 and 21 in a very convenient transmission mode.

1 Experimental

1.1 Materials

Single-side polished silicon wafer (<100>, *p*-type, boron-doped, 5.0 ~8.0 Ω·cm, 500 μm thick) was purchased from Hefei Kejing Materials Technology Co. Ltd. Silver nitrate was from Boshen Chemical Co. Ltd (Shanghai, China). *p*-ATP, *p*-nitrobenzoic acid, *p*-fluorothiophenol, thioglycolic acid and 11-mercaptoundecanoic acid were purchased from J&K Chemical Ltd, (China). Others were from Nanjing Chemical Co. Ltd (Nanjing, China).

1.2 Preparation of PSi

Single-side polished silicon wafer was cut into 1.8×1.8 cm² squares, cleaned by a boiling piranha solution ($V_{\text{H}_2\text{SO}_4}:V_{\text{H}_2\text{O}_2}=3:1$) for 30 min to remove organic contaminants (*Caution: Piranha solution is an extremely strong oxidant and should be handled very carefully!*). Rinsed with copious deionized water and absolute ethanol, silicon chips were stored in deionized water prior to etching.

The silicon chip was placed in a Teflon etching cell using a piece of aluminum foil as a back contact and a 1.4 cm diameter O-ring to seal the wafer to the cell. The cell was filled with a 3/1 (V/V) mixture of 40% aqueous HF and absolute ethanol. A platinum mesh was immersed in the solution as the anode. The etching process lasted for 10.0 min in dark with a constant anodic current density of 30.0 mA·cm⁻². After etching, the chip was removed from the cell, washed with absolute ethanol and dried under a stream of nitrogen.

1.3 SEIRA sample preparation and measurements

The freshly etched PSi chip was submerged in AgNO₃ aqueous solution for desired reaction time. The reduction-oxidation reaction happened immediately on the porous silicon surface and was summarized as $\text{SiH}_x + 2\text{Ag}^+ + 2\text{NO}_3^- + 3\text{H}_2\text{O} = \text{SiO}_2 + 2\text{Ag} + 2\text{H}^+ + 2\text{NO}_3^- + (1+x/2)\text{H}_2 \uparrow$ ^[33]. The Ag-NPs deposited PSi chip was rinsed with copious water and dried under a stream of nitrogen. To prepare the sample for SEIRA measurements, the probe molecule was dissolved in absolute ethanol in different concentrations (*p*-aminothiophenol, 10 mmol·L⁻¹; *p*-nitrobenzoic acid, 10 mmol·L⁻¹ and 100 mmol·L⁻¹; *p*-

fluorothiophenol (1 mL *p*-fluorothiophenol diluted by ethanol to 100 mL), $\sim 94 \text{ mmol} \cdot \text{L}^{-1}$), in order to obtain the optimum concentration for infrared enhancement and to achieve signals in control experiments on bare PSi chips. After the Ag-NPs/PSi chip was placed horizontally, 2.0 μL probe molecule solution was dropped on the 1.4 cm diameter round chip by microsyringe and dried spontaneously. Infrared spectra were recorded with a Bruker VERTEX 80V FTIR spectrometer at a resolution of 4 cm^{-1} for 32 scans. Cleaned single-side polished silicon wafer was used as background. The spectral baselines were corrected for clear demonstration. All spectra were disposed with the OPUS software and spectral windows were cut down to illuminate the significant changes. Field-emission scanning electron microscope (FE-SEM, Hitachi S-4800) with a point-to-point resolution of 2 nm was used to observe the morphology of samples at an accelerating voltage of 15.0 kV.

2 Results and discussion

2.1 Appropriate conditions for preparing Ag-NPs films on PSi substrates with *p*-ATP as the probe molecule

p-ATP was used as a probe molecule to assay the optimum silver deposition condition for SEIRA. The AgNO_3 concentration was optimized in the range of $2.5 \sim 5.0 \text{ mmol} \cdot \text{L}^{-1}$ after a series of experiments according to our previous reports^[34]. At an AgNO_3 concentration lower than $2.5 \text{ mmol} \cdot \text{L}^{-1}$, it needs more time to deposit Ag-NPs and at a concentration higher than $5.0 \text{ mmol} \cdot \text{L}^{-1}$ it leads to an uncontrollable deposition process. We prepared six Ag-NPs films on PSi chips after deposition for 1.0, 3.0, 5.0, 7.0, 8.0, and 9.0 min with $2.5 \text{ mmol} \cdot \text{L}^{-1} \text{AgNO}_3$ solution, respectively. As described in the experimental section, each chip has a circular shape with a diameter of 1.4 cm. We dropped 2.0 μL of $10 \text{ mmol} \cdot \text{L}^{-1}$ *p*-ATP ethanol solution on the chip placed horizontally and the ethanol solution spread homogeneously on the circular area and dried spontaneously within seconds, resulting in a homogeneous probe molecule film with a surface density of $13.0 \text{ nmol} \cdot \text{cm}^{-2}$. Fig.1 shows seven

transmission IR spectra of *p*-ATP on these Ag-NPs/PSi chips (Fig.1a to f) and on a bare PSi chip (Fig.1g) as reference. According to the surface selection rule, only those vibrational modes giving rise to an oscillating dipole perpendicular to the metal surface could be enhanced. Obviously three bands, 1 618, 1 590 and $1\,485 \text{ cm}^{-1}$ are enhanced in Fig.1b to f. As we previously assigned $1\,618 \text{ cm}^{-1}$ to NH_2 deformation, 1 590 and $1\,485 \text{ cm}^{-1}$ to the benzene ring skeletal stretching modes^[35], we can infer that the benzene ring stands nearly perpendicular to Ag-NPs. Taking the *p*-ATP spectrum in Fig.1g, recorded with the same surface density of $13.0 \text{ nmol} \cdot \text{cm}^{-2}$ on a bare PSi chip, as reference, we estimate the enhancement factors to be 2, 8, 9, 10, 4 and 2 for spectra a, b, c, d, e and f, respectively, based on the peak height at 1485 cm^{-1} . The above results reveal that Ag-NPs films deposited for 5.0~7.0 min provide the strongest enhancement. As time extends, the infrared enhancement is suppressed due to the evolution of silver nano-particle structures and partly the increasing blocking of infrared light. The similar enhancement trend can be observed at different AgNO_3 concentrations. However, the time window for the maximum enhancement is different, for example, the maximum enhancement for $5.0 \text{ mmol} \cdot \text{L}^{-1} \text{AgNO}_3$

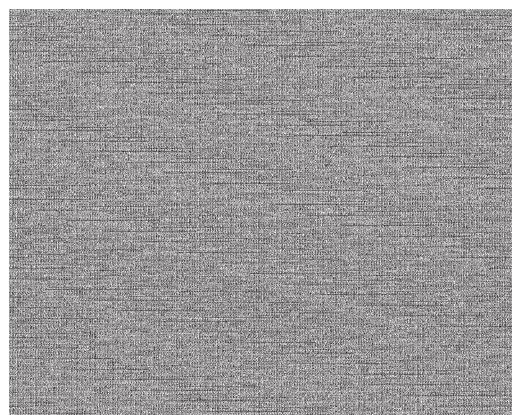


Fig.1 SEIRA spectra of *p*-ATP with a surface density of $13.0 \text{ nmol} \cdot \text{cm}^{-2}$ adsorbed on Ag-NPs/PSi substrates obtained by immersing freshly anodized PSi chips in $2.5 \text{ mmol} \cdot \text{L}^{-1} \text{AgNO}_3$ solution for (a) 1.0 min, (b) 3.0 min, (c) 5.0 min, (d) 7.0 min, (e) 8.0 min, and (f) 9.0 min. For convenience to compare the infrared enhancement effect, a reference spectrum of *p*-ATP with the same surface density adsorbed on a bare PSi chip is shown as (g)

solution can be achieved at about 2.0 min.

The different enhancement factors are of course associated with the surface structures of Ag-NPs films. As reported, SEIRA has only been observed on rough metal surfaces or metal island films^[2-3]. In the case of island films, the largest enhancement has been observed when the islands are densely crowded but not touching each other^[24-25]. We investigated the topographical structures of six Ag-NPs films used for SEIRA by SEM. The six images are shown in Fig.2. The films are composed of silver nano-particles. Obviously the particle size on the outmost layer becomes bigger as time going on. In Fig.2a and b, particles are flaky and streamline shaped, and the films are thin and flat. With time going on, particles in Fig.2c and d are more three-dimensional and crystal shaped, and the silver particulate films become thicker. The crystal shaped particles are averaged with a diameter of (135 ± 10) nm.

When the reaction time extends longer than 7.0 min, Fig.2e and f show bigger particles and longer distances from peak to valley. The increasing inhomogeneity of Ag-NPs over time should be the main factor for the suppression of SEIRA. Combination analyses of SEIRA in Fig.1 and the corresponding morphologies of Ag-NPs in Fig.2 suggest that metal particles with proper size, crystal shape, and aggregation with crowding but not connecting to each other play an important role in the enhancement. To further prove this statement, we eroded the Ag-NPs film of Fig.2d in 16% HNO_3 solution for 1.0 min and its corresponding image is shown in the inset of Fig.2d. Obviously the crowding and crystal shaped structures of Ag-NPs are destroyed and a laminar and round-edged film present. The same *p*-ATP film was prepared on the HNO_3 -eroded chip and no significant infrared enhancement was observed (data not shown).

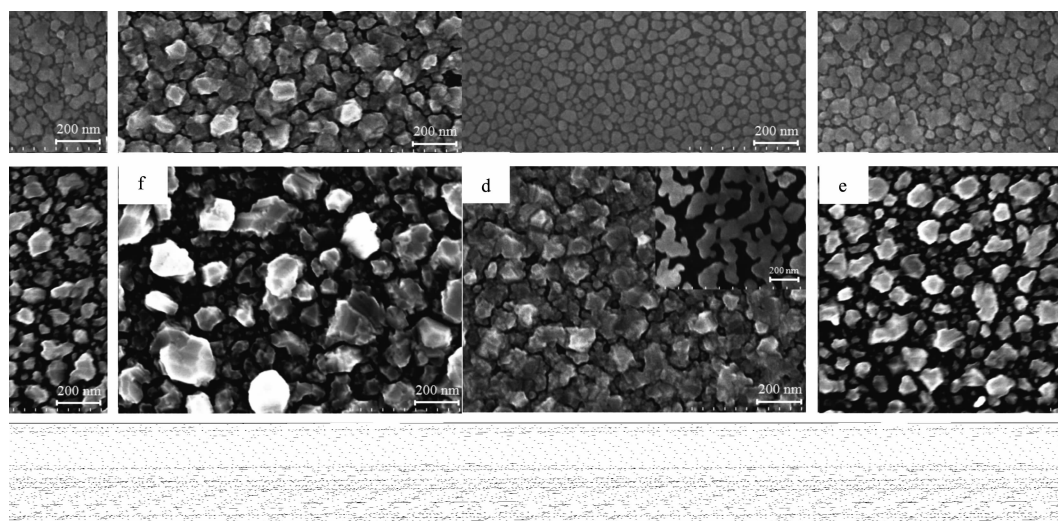


Fig.2 SEM images of Ag-NPs/PSi substrates obtained by immersing freshly anodized PSi chips in $2.5 \text{ mmol} \cdot \text{L}^{-1}$ AgNO_3 solution for (a) 1.0 min, (b) 3.0 min, (c) 5.0 min, (d) 7.0 min, (e) 8.0 min, and (f) 9.0 min. The inset in (d) is the SEM image of the same sample after eroding with 16% HNO_3 for 1.0 min

2.2 SEIRA spectra of *p*-nitrobenzoic acid and *p*-fluorothiophenol

Other two examples of SEIRA spectra are shown in Fig.3: *p*-nitrobenzoic acid in Fig.3A-b and *p*-fluorothiophenol in Fig.3B-b. For convenience to observe the enhancement effect directly, their corresponding spectra on bare PSi chips are also shown in Fig.3A-a and 3B-a respectively (note, to demonstrate the infrared bands clearly, the bottom

reference trace of *p*-nitrobenzoic acid in Fig. 3A-a is shown with a surface density of $130.0 \text{ nmol} \cdot \text{cm}^{-2}$, 10 times of *p*-nitrobenzoic acid quantities adsorbed on Ag-NPs/PSi). For *p*-nitrobenzoic acid in Fig.3A-b, its strong anti-symmetric stretching band of COOH at 1702 cm^{-1} on PSi disappears, instead a strong symmetric stretching band of COO^- at 1385 cm^{-1} and a medium asymmetric one at 1630 cm^{-1} appear, which proves the formation of $\text{COO}^- \text{Ag}^+$ bond. The most

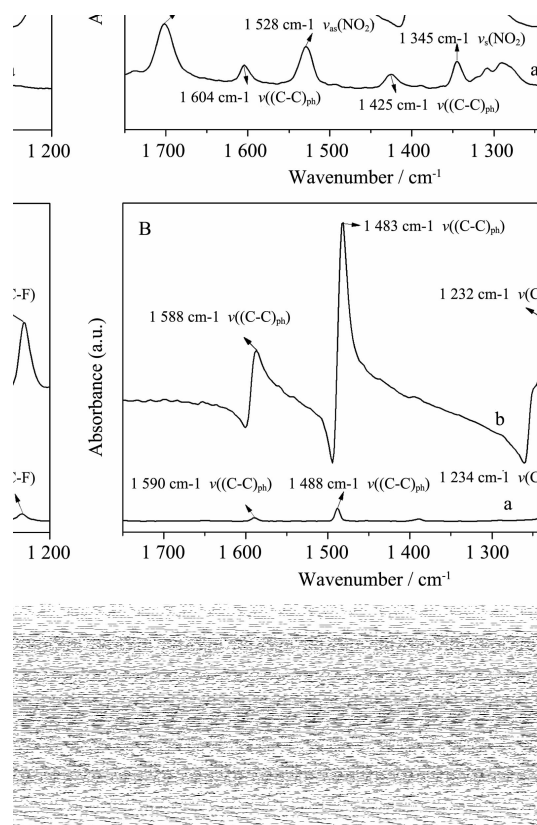


Fig.3 SEIRA spectrum of *p*-nitrobenzoic acid (upper trace in A-b) with a surface density of 13.0 nmol·cm⁻² and a reference spectrum of *p*-nitrobenzoic acid with a surface coverage of 130.0 nmol·cm⁻² (10 times surface density as in A-b for a clear demonstration of the spectrum) on PSi in the bottom trace of A-a; SEIRA spectrum of *p*-fluorothiophenol (upper trace in B-b) with a surface density of 122.0 nmol·cm⁻² and a reference spectrum of *p*-nitrobenzoic acid on PSi with the same surface density in the bottom trace of B-a

significant enhancement with 85 times magnification can be seen at 1 342 cm⁻¹, which is assigned to the symmetric stretch of NO₂. The last but not the least one, 122.0 nmol·cm⁻² *p*-fluorothiophenol in Fig.3B-b, exhibits the strongest benzene ring skeletal stretchings at 1 483 cm⁻¹ and 1 588 cm⁻¹. Its C-F stretching at 1 232 cm⁻¹ is also enhanced remarkably (21 times). The previously reported band asymmetry in SEIRA is also observed in all three bands^[36-37]. The “Fano band” distortion possibly links to the dynamic interaction of the adsorbate vibrations with electron-hole pair excitations^[38].

2.3 Discussion

Two enhancement mechanisms are generally accepted for SEIRA: (1) Electromagnetic (EM) enhancement^[2-3] is based on the hypothesis that the superposition of the incident and scattered fields interacts with the transition moment of a molecule near the metal surface. Localized surface plasmon resonance near the surface of a conducting sphere provides a means for amplification of EM fields, while the surface selection rule determines which vibration mode is enhanced^[2-3]. (2) Chemical enhancement relates to tight chemical binding of the molecule onto a metal surface^[2] and charge transfer between the energy levels of the molecule and Fermi levels of the metal, which result in reorganization of the molecule on the surface and increment of the polarizability of the adsorbate. Although the two mechanisms do not work separately, the first is considered as the essential one in most works and the second makes assistant contribution. In our experiments, we observed significant SEIRA phenomena on three model molecules, but we also failed to get the SEIRA spectra for other two molecules, thioglycolic acid and 11-mercaptoundecanoic acid. To analyze the SEIRA mechanisms in our case, we address two points. The first is the tight binding of a rigid molecule to Ag-NPs. For example both *p*-APT and *p*-fluorothiophenol can form a strong covalent bond of S-Ag with Ag-NPs and then they should be reorganized to stand nearly perpendicular with the longitudinal rigid benzene ring to the tangent of Ag-NPs, which generates significant enhancement of the skeletal stretching bands. The binding of *p*-nitrobenzoic acid to Ag-NPs through COO⁻-Ag is not as strong as S-Ag, however the strong enhancement of the -NO₂ symmetric stretch implies that the benzene ring stands straightly on Ag-NPs. The second is the charge transfer and increment of the polarizability (or separation of charges). *p*-Nitrobenzoic acid and *p*-fluorothiophenol possess strong electron-withdrawn groups, NO₂ and F, and thus abstract more electrons from Ag-NPs through the *d*-π conjugation and present significant infrared enhancement in NO₂ and C-F. Charge transfer also occurs on *p*-APT via the *d*-π conjugation, which can be

inferred from the band red-shift of benzene ring skeletal stretchings from porous silicon at $1\,493\text{ cm}^{-1}$ in Fig.1g to $1\,485\text{ cm}^{-1}$ on Ag-NPs in Fig.1b to f.

The failure of SEIRA measurements for two alkyl chain molecules of thioglycolic acid and 11-mercaptopundecanoic acid can be explained as follows: even with the strong binding group of -SH, (1) their alkyl chains are too flexible to stand straightly on the surface; (2) charge transfer cannot occur through the σ bond. The above results and statements agree well with the electromagnetic surface selection rule, that the polarizability tensor elements are perpendicular to a metal surface should be much more enhanced in SEIRA than parallel ones.

3 Conclusions

To summarize, we report a very convenient method to execute SEIRA measurements on probe molecule/Ag-NPs/PSi chips through transmission mode. Excellent enhancement performance as high as 85 times magnification can be achieved. The chip was fabricated through chemical deposition of Ag-NPs films onto the porous silicon membrane by reducing Ag^+ from AgNO_3 solution with surface SiH_x ($x=1, 2, 3$) species. Three molecules, *p*-ATP, *p*-nitrobenzoic acid and *p*-fluorothiophenol, were used as probes to study the SEIRA phenomena of Ag-NPs/PSi substrates. An optimum deposition condition of Ag-NPs was assayed by *p*-ATP as $2.5\text{ mmol}\cdot\text{L}^{-1}\text{ AgNO}_3$ for 5.0~7.0 min. SEM imaging reveals that $(135\pm10)\text{ nm}$ diameter nubby nanoparticles with interstitial grainy ones can lead to the strongest enhancement. The absence of any adjunctive substance on Ag-NPs during deposition, such as surfactants for capping, is of great benefit for SEIRA measurements. The naked Ag-NPs/PSi chip is also ideal for analyte adsorption and noise signal elimination. Molecules with dipole moment derivative components perpendicular to Ag-NPs are preferentially enhanced. Significant enhancement is observed when molecules have benzene ring with strong electron-withdrawn groups. Such a simple substrate benefit from the high specific surface area and infrared transparency of porous silicon will gain more attention for SEIRA

measurements.

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中国化学会第 28 届学术年会第一轮通知

中国化学会第 28 届学术年会将于 2012 年 4 月 13—16 日在四川省成都市举行。本届年会由中国化学会主办,四川大学承办,会期 4 天,4 月 12 日报到。2012 年正值中国化学会成立八十周年,届时将举办相关庆祝活动。

本届年会设立学术分会 19 个、专题论坛 4 个。学术交流形式包括大会特邀报告(PL)、分会邀请报告(I)、口头报告(O)和墙报(P)。年会面向全国广大化学工作者征集学术论文(摘要),并印制论文摘要集,欢迎踊跃投稿,积极参会。

年会期间将举办新技术、新产品与新仪器成果展览以及人才交流活动,欢迎相关企业、高校、科研院所积极参与。

会议详细内容请见会议网站 <http://www.ccs.ac.cn/confab/huiyi.html>。

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1.撰写论文摘要登录中国化学会网站(<http://www.ccs.ac.cn>),下载年会论文摘要模板,并根据模板要求撰写摘要。

2.会议注册登录中国化学会网站(<http://www.ccs.ac.cn>),正确填写参会信息,注册参会。

会议注册时间:2011 年 9 月 1 日—2012 年 2 月 29 日

3.递交论文摘要选择分会,并上传论文摘要。接受原创性、创新性研究论文和前瞻性综述(特邀)。

论文接收起止期:2011 年 10 月 1 日—2012 年 1 月 31 日