
Catalysis of atomic hydrogen to new hydrides as a new power source

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Abstract: Having the potential for a clean new energy source, rt-plasmas of certain catalysts (Sr⁺, Ar⁺, K) with H formed at extraordinary low field strengths of about 1–2 V/cm. Time-dependent, extraordinarily fast H (25 eV), an excess power of 20 mW · cm⁻³, and characteristic K³⁺ emission confirmed the resonant nonradiative energy transfer of 3 · 27.2 eV from atomic hydrogen to K as the rt-plasma catalyst. The predicted very stable novel hydride ion H(1/4) with fractional principal quantum number $p=4$ was observed spectroscopically at 110 nm corresponding to its predicted binding energy of 11.2 eV that further matched the ¹H MAS NMR spectrum having an extraordinary upfield-shifted peak at -4.4 ppm with the elimination of any known assignment by FTIR.

Keywords: H catalysis; fast H; exothermic; novel hydride ions; upfield NMR peaks; FTIR.

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1 Introduction

A new chemically generated or assisted plasma source based on a resonant energy transfer mechanism (rt-plasma) has been developed that may be a new power source. One such source operates by incandescently heating a hydrogen dissociator and a catalyst to provide atomic hydrogen and gaseous catalyst, respectively, such that the catalyst reacts with the atomic hydrogen to produce a plasma. It was extraordinary that intense Extreme Ultraviolet (EUV) emission was observed by Mills et al. (2000a, 2002a, 2002b, 2003a, 2004) and Conrads et al. (2003) at low temperatures (e.g., $\approx 10^3$ K) and an extraordinary low field strength of about 1–2 V/cm from atomic hydrogen and certain atomised elements or certain gaseous ions which singly or multiply ionise at integer multiples of the potential energy of atomic hydrogen, 27.2 eV. A number of independent experimental observations confirm that the rt-plasma is due to a novel reaction of atomic hydrogen which produces as chemical intermediates, hydrogen in fractional quantum states that are at lower energies than the traditional 'ground' ($n = 1$) state. Power is released (Mills et al., 2003b, 2004; Phillips et al., 2004), and the final reaction products are novel hydride compounds (Mills et al., 2000b, 2001a, 2001b, 2004) as reported herein or lower-energy molecular hydrogen (Mills et al., 2005a). The supporting data include EUV spectroscopy (Mills, 2001; Mills and Ray, 2002a, 2002b, 2003a, 2003b, 2003c; Mills et al., 2000a, 2002a, 2002b, 2002c, 2003a, 2003b, 2003d, 2004, 2005b; Conrads et al., 2003), characteristic emission from catalysts and the hydride ion products (Mills, 2001; Mills and Ray, 2003b, 2003c; Mills et al., 2002b, 2003a, 2003d, 2004), lower-energy hydrogen emission (Mills and Ray, 2002a, 2003a; Mills et al., 2002c, 2003b), chemically formed plasmas (Mills, 2001; Mills and Ray, 2003b, 2003c; Mills et al., 2000a, 2002a, 2002b, 2003a, 2003d, 2004; Conrads et al., 2003), extraordinary (>100 eV) Balmer α line broadening (Mills and Ray, 2002b, 2003a, 2003b, 2003c; Mills et al., 2002a, 2002b, 2002c, 2002d, 2003a, 2003b, 2003c, 2003d, 2004, 2005b, 2006), population inversion of H lines (Mills and Ray, 2003b, 2003c; Mills et al., 2003d, 2003e,

2004, 2005b), elevated electron temperature (Mills and Ray, 2002b; Mills et al., 2002c, 2002d, 2003c), anomalous plasma afterglow duration (Mills et al., 2004, Conrads et al., 2003), power generation (Mills et al., 2003b, 2004; Phillips et al., 2004), and analysis of novel chemical compounds (Mills et al., 2000b, 2001a, 2001b, 2004).

The theory given previously (Mills, 2006; Mills and Ray, 2002a, 2003a; Mills et al., 2000a, 2002c) is based on applying Maxwell's equations to the Schrödinger equation. The familiar Rydberg equation (1) arises for the hydrogen excited states for $n > 1$ of equation (2).

$$E_n = -\frac{e^2}{n^2 8\pi\epsilon_0 a_H} = -\frac{13.598 \text{ eV}}{n^2} \quad (1)$$

$$n = 1, 2, 3, \dots \quad (2)$$

An additional result is that atomic hydrogen may undergo a catalytic reaction with certain atoms, excimers, and ions which provide a reaction with a net enthalpy of an integer multiple of the potential energy of atomic hydrogen, $m \cdot 27.2 \text{ eV}$ wherein m is an integer. The reaction involves a nonradiative energy transfer to form a hydrogen atom that is lower in energy than unreacted atomic hydrogen that corresponds to a fractional principal quantum number. That is

$$n = \frac{1}{2}, \frac{1}{3}, \frac{1}{4}, \dots, \frac{1}{p}; \quad p \text{ is an integer} \quad (3)$$

replaces the well known parameter $n = \text{integer}$ in the Rydberg equation for hydrogen excited states. The $n = 1$ state of hydrogen and the $n = 1/\text{integer}$ states of hydrogen are nonradiative, but a transition between two nonradiative states, say $n = 1$ to $n = 1/2$, is possible via a nonradiative energy transfer. Thus, a catalyst provides a net positive enthalpy of reaction of $m \cdot 27.2 \text{ eV}$ (i.e., it resonantly accepts the nonradiative energy transfer from hydrogen atoms and releases the energy to the surroundings to affect electronic transitions to fractional quantum energy levels). As a consequence of the nonradiative energy transfer, the hydrogen atom becomes unstable and emits further energy until it achieves a lower-energy nonradiative state having a principal energy level given by equations (1) and (3). Processes such as hydrogen molecular bond formation that occur without photons and that require collisions are common (Sidgwick, 1950). Also, some commercial phosphors are based on resonant nonradiative energy transfer involving multipole coupling (Lamb, 1978).

He^+ fulfills the catalyst criterion – a chemical or physical process with an enthalpy change equal to an integer multiple of 27.2 eV since it ionises at 54.417 eV which is $2 \cdot 27.2 \text{ eV}$. Ar^+ may also serve as a catalyst since its ionisation energy is about 27.2 eV. The product of the catalysis reaction of He^+ , $\text{H}(1/3)$, may further serve as a catalyst to form $\text{H}(1/4)$ and $\text{H}(1/2)$ (Mills, 2006; Mills and Ray, 2002a; Mills et al., 2002c). The product of the Ar^+ catalysis reaction, $\text{H}(1/2)$, may further serve as both a catalyst and a reactant to form $\text{H}(1/4)$ (Mills, 2006; Mills and Ray, 2002a; Mills et al., 2002a, 2002b, 2002c, 2003a). Novel emission lines with energies of $q \cdot 13.6 \text{ eV}$ where $q = 1, 2, 3, 4, 6, 7, 8, 9$ or 11 were previously observed by EUV spectroscopy recorded on microwave discharges of helium with 2% hydrogen (Mills and Ray, 2002a; Mills et al., 2002c). These lines matched $\text{H}(1/p)$, fractional Rydberg states of atomic hydrogen wherein

$$n = \frac{1}{2}, \frac{1}{3}, \frac{1}{4}, \dots, \frac{1}{p}; (p \leq 137 \text{ is an integer})$$

replaces the well known parameter $n = \text{integer}$ in the Rydberg equation for hydrogen excited states. Two $\text{H}(1/p)$ may react to form $\text{H}_2(1/p)$ that has vibrational and rotational energies that are p^2 times those of H_2 comprising uncatalysed atomic hydrogen (Mills, 2004, 2006). Rotational lines were observed in the 145–300 nm region from atmospheric pressure electron-beam excited argon-hydrogen plasmas. The unprecedented energy spacing of 4^2 times that of hydrogen established the internuclear distance as $1/4$ that of H_2 and identified $\text{H}_2(1/4)$. $\text{H}_2(1/p)$ gas was isolated by liquefaction using an high-vacuum (10^{-6} Torr) capable, liquid nitrogen cryotrap and was characterised by Mass Spectroscopy (MS). The condensable gas had a higher ionisation energy than H_2 by MS (Mills et al., 2005a).

Water bath calorimetry was used to demonstrate one more peculiar phenomenon associated with rt-plasmas. Specifically, He/H_2 (10%) (500 mTorr), Ar/H_2 (10%) (500 mTorr), and $\text{H}_2\text{O}(g)$ (500 mTorr and 200 mTorr) plasmas generated with an Evenson microwave cavity consistently yielded on the order of 50% more heat than non rt-plasma (controls) such as He, Kr, Kr/H_2 (10%), under identical conditions of gas flow, pressure, and microwave operating conditions. The excess power density of rt-plasmas was of the order $10 \text{ W} \cdot \text{cm}^{-3}$. In addition to unique Vacuum Ultraviolet (VUV) lines, earlier studies with these same rt-plasmas demonstrated that other unusual features were present including dramatic broadening of the hydrogen Balmer series lines (Mills and Ray, 2002b, 2003a, 2003b, 2003c; Mills et al., 2002a, 2002b, 2002c, 2002d, 2003a, 2003b, 2003c, 2003d, 2004, 2005b, 2006), and in the case of water plasmas, population inversion of the hydrogen excited states (Mills and Ray, 2003b, 2003c; Mills et al., 2003d, 2003e, 2004, 2005b). Both the current results and the earlier results are completely consistent with the existence of a hitherto unknown predicted exothermic chemical reaction occurring in rt-plasmas.

Since the ionisation energy of Sr^+ to Sr^{3+} has a net enthalpy of reaction of $2 \cdot 27.2 \text{ eV}$, respectively, Sr^+ may serve as catalyst alone or with Ar^+ catalyst. It was reported previously that an rt-plasma formed with a low field (1 V/cm), at low temperatures (e.g., $\approx 10^3 \text{ K}$), from atomic hydrogen generated at a tungsten filament and strontium which was vapourised by heating the metal (Mills et al., 2002a, 2002b, 2003a). Strong VUV emission was observed that increased with the addition of argon, but not when sodium, magnesium, or barium replaced strontium or with hydrogen, argon, or strontium alone. Characteristic emission was observed from a continuum state of Ar^{2+} at 45.6 nm without the typical Rydberg series of Ar I and Ar II lines which confirmed the resonant nonradiative energy transfer of 27.2 eV from atomic hydrogen to Ar^+ (Mills, 2001; Mills et al., 2002b, 2003a). Predicted Sr^{3+} emission lines were also observed from strontium-hydrogen plasmas (Mills et al., 2002b, 2003a) that supported the rt-plasma mechanism.

Significant Balmer α line broadening corresponding to an average hydrogen atom temperature of 14, 24 eV, and 23–45 eV was observed for strontium and argon-strontium rt-plasmas and discharges of strontium-hydrogen, helium-hydrogen, argon-hydrogen, strontium-helium-hydrogen, and strontium-argon-hydrogen, respectively, compared to $\approx 3 \text{ eV}$ for pure hydrogen, krypton-hydrogen, xenon-hydrogen, and magnesium-hydrogen. To achieve that same optically measured light output power, hydrogen-sodium, hydrogen-magnesium, and hydrogen-barium mixtures required 4000, 7000 and 6500

times the power of the hydrogen-strontium mixture, respectively, and the addition of argon increased these ratios by a factor of about two. A glow discharge plasma formed for hydrogen-strontium mixtures at an extremely low voltage of about 2 V compared to 250 V for hydrogen alone and sodium-hydrogen mixtures, and 140–150 V for hydrogen-magnesium and hydrogen-barium mixtures (Mills et al., 2002a, 2002b, 2003a). These voltages are too low to be explicable by conventional mechanisms involving accelerated ions with a high applied field.

To further characterise argon-strontium rt-plasmas, plasma formation was studied relative to mixtures of hydrogen and a chemically similar control that does not have electron ionisation energies which are a multiple of 27.2 eV, and the Balmer lines were recorded by visible spectroscopy to confirm that an energetic hydrogen plasma was present having H energy states greater than 12 eV corresponding to $n \geq 3$ in equations (1) and (2). The broadening of the Balmer α line was also recorded as a function of time, and thermal power balance measurements were performed. The cell comprised a titanium or tungsten filament to heat and vapourise some strontium as a source of catalyst and to dissociate molecular hydrogen to atomic hydrogen. The addition of argon to the plasma further provided the catalyst Ar^+ .

Since a conventional discharge power source was not present, the formation of a plasma would require an energetic reaction. The origin of Doppler broadening is the relative thermal motion of the emitter with respect to the observer – in this case the spectrometer. Line broadening is a measure of the atom temperature, and a significant increase was expected and observed for catalysts, strontium or argon, with hydrogen. The observation of a high hydrogen temperature with no conventional explanation would indicate that an rt-plasma must have a source of free energy. An energetic chemical reaction was further implicated since it was found that the broadening is time dependent. Therefore, the thermal power balance was measured calorimetrically. We report the results of these characterisations and discuss the implications regarding the rt-plasma mechanism in Sections 3.1–3.3.

2K^+ to $\text{K}+\text{K}^{2+}$ and K to K^{3+} also provide a reaction with a net enthalpy equal to one and three times the potential energy of atomic hydrogen, respectively. It was reported previously (Mills et al., 2004) that the presence of these gaseous ions or atoms with thermally dissociated hydrogen formed an rt-plasma having strong VUV emission with a stationary inverted Lyman population. Significant line broadening of the Balmer α , β , and γ lines of 18 eV was observed, compared to 3–4 eV from a hydrogen microwave plasma. Emission from rt-plasmas occurred even when the electric field applied to the plasma was zero. The reaction was exothermic since excess power of $20 \text{ mW} \cdot \text{cm}^{-3}$ was measured by Calvet calorimetry. An energetic catalytic reaction was proposed involving a resonant energy transfer between hydrogen atoms and 2K^+ or K to form very stable novel hydride ions $\text{H}^-(1/p)$ called hydrino hydrides having a fractional principal quantum numbers $p = 2$ and $p = 4$, respectively. Characteristic emission was observed from K^{2+} and K^{3+} that confirmed the resonant nonradiative energy transfer of 27.2 eV and $3 \cdot 27.2 \text{ eV}$ from atomic hydrogen to 2K^+ and K , respectively. The product hydride ion $\text{H}^-(1/4)$ was observed spectroscopically at 110 nm corresponding to its predicted binding energy of 11.2 eV. The ^1H MAS NMR spectrum of novel compound KH^*Cl relative to external tetramethylsilane (TMS) showed a large distinct upfield resonance at -4.4 ppm corresponding to an absolute resonance shift of -35.9 ppm that matched the theoretical prediction of $p = 4$. A novel peak of KH^*I at -1.5 ppm relative to TMS corresponding to

an absolute resonance shift of -33.0 ppm matched the theoretical prediction of $p = 2$. The predicted catalyst reactions, position of the upfield-shifted NMR peaks for $\text{H}^-(1/4)$ and $\text{H}^-(1/2)$, and spectroscopic data for $\text{H}^-(1/4)$ were found to be in agreement with the experimental observations as well as previously reported spectroscopic data for $\text{H}^-(1/2)$ and analysis of KH^*Cl and KH^*I containing these hydride ions.

The upfield-shifted NMR peaks are a direct evidence of the existence of lower-energy state hydrogen with a reduced radius relative to ordinary hydride ion and having an increase in diamagnetic shielding of the proton. The total theoretical shift $\Delta B_T/B$ for $\text{H}^-(1/p)$ is given by the sum of the shift of $\text{H}^-(1/1)$ plus the contribution due to the lower-electronic energy state:

$$\frac{\Delta B_T}{B} = -\mu_0 \frac{e^2}{12m_e a_0 (1 + \sqrt{s(s+1)})} (1 + \alpha 2\pi p) = -(29.9 + 1.37p) \text{ ppm} \quad (4)$$

where $p = \text{interget} > 1$. Corresponding alkali hydrides and alkali hydrino hydrides (containing $\text{H}^-(1/p)$) were characterised by ^1H MAS NMR and compared to the theoretical values. A match of the predicted and observed peaks with no alternative represents a definite test. Elemental analysis identified (Mills et al., 2000b, 2001a) these compounds as only containing the alkaline metal, halogen, and hydrogen, and no known hydride compound of this composition could be found in the literature which has an upfield-shifted hydride NMR peak. Ordinary alkali hydrides alone or mixed with alkali halides show down-field shifted peaks (Mills et al., 2000b, 2001a, 2001b, 2004).

From the literature, the list of alternatives to $\text{H}^-(1/p)$ as a possible source of the upfield NMR peaks is limited. Although, the candidates, F-centres (an electron bound to an anion vacancy) and substitution H^- ions (U centres) in alkali halide crystals have been a substantial research subject in the literature due to potential optical properties. These ionic crystals were normally prepared by reaction of alkali halide with hydrogen under high temperature and high pressure conditions with further aide of UV or laser irradiation. The NMR spectrum of U centres are not known, and typically they are only found in trace amounts in alkali halides. Thus, NMR may not be sensitive enough to record their spectrum. However, the H^- substituted alkali halide crystals exhibit a distinguishable UV absorption bands (U) in various compounds. Infrared spectroscopic study of the vibration band of substitution H^- reveals peak positions for KCl, KBr, KI, and RbI at 503 , 446 , 382 and 478 cm^{-1} , respectively (Abeles, 1972). The intense and characteristic infrared spectra enabled us to study the relationship between the upfield-shifted NMR peaks any U centred H that may somehow have caused the upfield-shifted peaks. By performing FTIR as well as NMR on samples, we found that the novel NMR peaks did not belong to U centred H. This result supports previous observations from the rt-plasmas of intense hydrogen Lyman emission, a stationary inverted Lyman population, excessive afterglow duration, highly energetic hydrogen atoms, characteristic alkali-ion emission due to catalysis, predicted novel spectral lines, and the measurement of a power beyond any conventional chemistry (Mills et al., 2004). These observations matched predictions for a catalytic reaction of atomic hydrogen to form more stable hydride ions designated $\text{H}^-(1/p)$. We report the results of these characterisations and discuss the implications regarding the rt-plasma reaction product in Section 3.4. Section 4 gives a summary.

2 Experimental

An argon-hydrogen (97/3%)-strontium rt-plasma was generated in the experimental set up described previously (Mills et al., 2002a, 2002b, 2003a, 2004) comprising a thermally insulated quartz cell with a cap that incorporated ports for gas inlet, and outlet. A titanium filament (55 cm long, 0.5 mm diameter) that served as a heater and hydrogen dissociator was in the quartz tube. 2.5 g of magnesium or strontium metal (Alfa Aesar 99.95%) was placed in the centre of the cell under one atmosphere of dry argon in a glovebox. The cell was sealed and removed from the glovebox. The cell was maintained at 50°C for four hours with helium flowing at 30 sccm at a pressure of 0.6 Torr. The filament power was increased to 120 W in 20 increments every 20 minutes. At 120 W, the filament temperature was estimated to be in the range 800–1000°C. The external cell wall temperature was about 700°C. The cell was then operated with and without an argon-hydrogen (90/10%) flow rate of 5.5 sccm maintained at 0.6 Torr. Additionally, the cell was operated with hydrogen and argon-hydrogen (90/10%) gas flow and no metal. Each metal was vapourised by the filament heater. The presence of a hydrogen plasma was determined by recording the visible spectrum over the Balmer region with a Jobin Yvon Horiba 1250 M spectrometer with a PMT detector described previously (Mills et al., 2002b, 2002d, 2003c) using entrance/exit slits of 200/100 μm , 0.1 Å step size, and a 3 s integration time. The width of the 656.3 nm Balmer α line emitted from the argon-hydrogen (90/10%)-strontium rt-plasma having a titanium filament was measured initially and periodically during operation.

The power balance of a rt-plasma of strontium with argon-hydrogen mixture (95/5%) was measured with the experimental setup shown in Figure 1. The power balances of argon-hydrogen-strontium rt-plasmas maintained in a one-litre cylindrical stainless steel cell fitted with a heated tungsten filament shown in Figure 2 were measured by heat loss calorimetry (determining the power balance from the temperature at steady state relative to that of a control power source) as the input power to the filament was varied. The relationship between the rate of heat loss from the cell and the cell temperature was determined from a control experiment in which both strontium catalyst and plasma were absent.

The 304-stainless steel cylindrical cell was 9.21 cm in diameter and 14.5 cm in height. The base of the cell contained a welded-in stainless steel thermocouple well (1 cm OD) which housed a thermocouple in the cell interior approximately 3 cm from the cell axis. The upper end of the cell was welded to a high vacuum 15 cm diameter conflat flange. A silver-plated copper gasket was used to seal the cell flange to a mating flange. The two flanges were clamped together with 10 circumferential bolts. The mating flange contained two penetrations comprising a stainless steel thermocouple well (1 cm OD), which also housed a thermocouple in the cell interior approximately 3.5 cm from the cell axis, and a centred high voltage feed through. The body of the cell included two radial penetrations. One was a 9.5 mm OD tube for gas fill and evacuation, the other was a tube which housed a 1.5 cm diameter UV-grade sapphire viewport. The cell interior was fitted with a 27 mm OD grooved alumina tube which was 60 mm in length. The tube was tightly wrapped with approximately 330 cm of 0.25 mm tungsten wire. The tube was suspended on the cell axis by connecting the ends of the tungsten filament to the central lead and the cell body (ground) as shown in Figure 2. AC power at 60 Hz was supplied to the filament through a variac. True rms voltage and current, and also power dissipation in the filament were monitored by a digital volt-amp-watt meter (Clarke-Hess Model 259

V-A-W Meter) shown in Figure 1. Gas pressure in the cell was monitored with a 0–10 Torr MKS Baratron absolute pressure gauge.

Figure 1 The experimental setup for generating a argon-hydrogen-strontium rt-plasma and for measuring the power balance

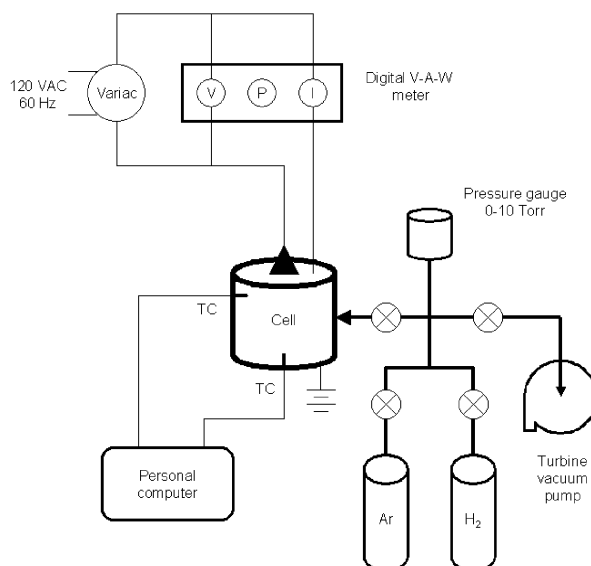
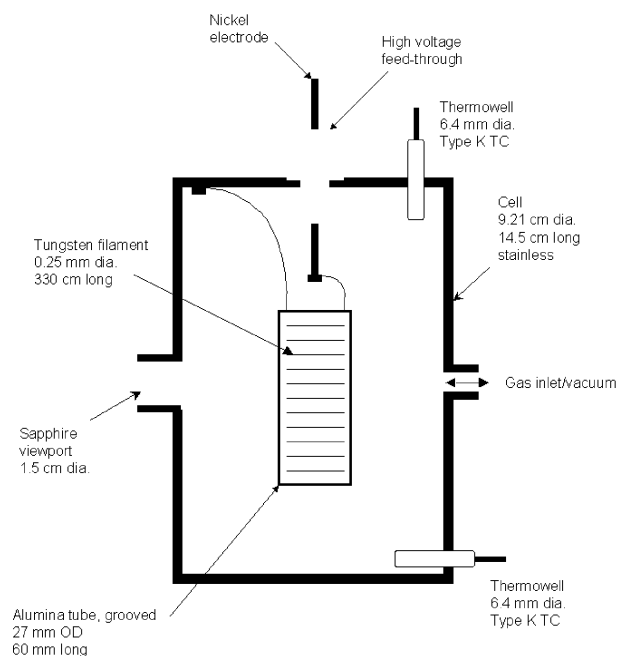


Figure 2 The one-litre cylindrical stainless steel cell fitted with a heated tungsten filament used to measure the power balances of argon-hydrogen-strontium rt-plasmas as a function of input power to the filament



Approximately 1.2 g of strontium metal (Alfa Aesar 99.95%) was placed on the base of the cell under one atmosphere of dry argon in a glovebox. After sealing the cell it was heated in a temperature-controlled kiln to 465°C while evacuating the system. At this condition the strontium vapour pressure was approximately 1 mTorr. After reaching thermal equilibrium, the cell was pressurised with 190 mTorr of argon and then an additional 10 mTorr of hydrogen to yield a argon-hydrogen mixture (95/5%) at 200 mTorr. Hydrogen was periodically added during the course of the experiment in order to maintain 200 mTorr pressure. It was observed that a substantial amount of hydrogen was absorbed during addition which was attributed to formation of strontium hydride. Ultrahigh purity grade argon and hydrogen were used. Approximately 50 V was applied to the filament corresponding to about 100 W. Plasma formation resulted after several minutes of heating the cell with the filament. Strong strontium and argon plasma line emission were observed in the visible and near-infrared with a visible spectrometer (Ocean Optics S2000). Electric power input to the cell was varied in the range 110–245 W. At each power setting, 2.5–3 hours was allowed for the cell to reach thermal equilibrium. The cell temperature was then computed by averaging the temperatures of the two thermocouples. In the control experiment, this procedure was repeated with the same cell except that both strontium and the resulting rt-plasma were absent.

Potassium chloro hydride (KH*Cl) was synthesised by reaction of atomic hydrogen with potassium metal (Aldrich Chemical Company 99%) as the catalyst with the corresponding alkali halide KCl (Alfa Aesar ACS grade 99+%) as an additional reactant. The compound was prepared in a stainless steel gas cell comprising a Ni screen hydrogen dissociator (Belleville Wire Cloth Co., Inc.), catalyst, and alkali halide as described previously (Mills et al., 2000b, 2001a, 2001b). The reactor was run at 650°C in a kiln for 72 hours, then cooled under helium atmosphere. The sealed reactor was then opened in the environmental chamber. NMR samples were placed in glass ampules, sealed with a rubber septa, and transferred out of the chamber to be flame sealed in atmosphere.

¹H MAS NMR was performed on solid samples of KH*Cl at Spectral Data Services, Inc., Champaign, Illinois as described previously (Mills et al., 2000b, 2001a, 2001b, 2004). The samples were handled under a inert atmosphere. Chemical shifts were referenced to external TMS. To eliminate the possibility that the alkali halide KCl influenced the local environment of the ordinary alkali hydride KH to produce an NMR resonance that was shifted upfield relative to KH alone, controls comprising KH and an equimolar KH/KCl mixture were run. The reference of each novel hydride comprised the corresponding ordinary hydride KH (Aldrich Chemical Company 99%) and equivalent molar mixtures of KH and KCl prepared in a glove box under argon.

FTIR analysis was performed on solid sample pellets using the transmittance mode at Department of Chemistry, Princeton University, New Jersey using a Nicolet 730 FTIR spectrometer with DTGS detector at resolution of 4 cm⁻¹. The instrument was equipped with a dry nitrogen purge at ambient temperature. During the data acquisition, about 640–1024 scans were obtained. IR pellets were prepared in the environmental chamber by grinding the KH*Cl solid sample to a fine powder using a mortar and pestle and mixing it with fine KBr powder (FTIR Spectra Grade, International Crystal Lab, Inc. NJ) at a volumetric ratio of sample to KBr of about 1 : 1. The mixed powder was further ground and uniformly mixed, then sprinkled into a 13 mm die to make an IR transparent pellet with about 10 tons of pressure using a self-contained hydraulic jack. A paper insert with 11.0 mm diameter aperture (International Crystal Lab) was used in the pellet making. The paper insert did not affect the spectroscopic measurement. The transparent

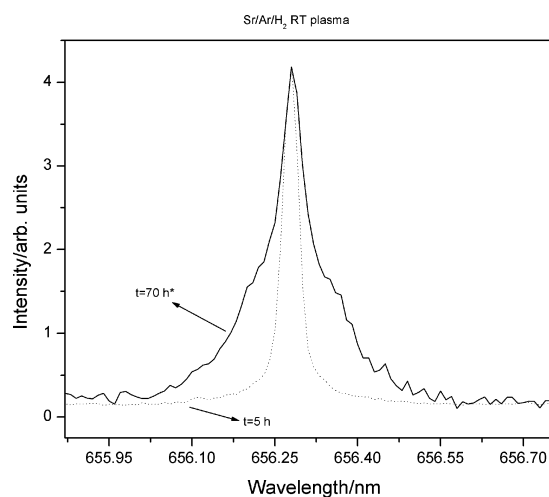
pellet was held within the aperture (~ 13.5 mm) in a Teflon sheet, which was placed in between two KBr windows (32×3 mm) and sealed by a Viton O-ring (32 mm diameter) to avoid any air leak. The whole assembly was packed in an aluminum sample holder (SL-2 cell kit, International Crystal Lab).

3 Results and discussion

3.1 RT-plasma emission

An argon-hydrogen (90/10%)-strontium rt-plasma formed with a low field (1 V/cm), at low temperatures (e.g., $\approx 10^3$ K), from atomic hydrogen generated at a titanium filament and strontium which was vapourised by heating the metal. H Balmer emission corresponding to population of a level with energy >12 eV was observed as shown in Figure 3 which also requires that Lyman emission was present. No plasmas formed when magnesium replaced strontium or with hydrogen, argon/hydrogen, or strontium alone. This result indicates that the emission was due to a reaction of hydrogen with vapourised strontium. No possible chemical reactions of the titanium filament, the vapourised strontium, and 0.6 Torr argon-hydrogen mixture at a cell temperature of 700°C could be found which accounted for the Balmer emission. In fact, no known chemical reaction releases enough energy to excite Balmer and Lyman emission from hydrogen. In addition to known chemical reactions, electron collisional excitation, resonant photon transfer, and the lowering of the ionisation and excitation energies by the state of 'non ideality' in dense plasmas were also rejected as the source of ionisation or excitation to form the hydrogen plasma (Conrads et al., 2003). The formation of an energetic reaction of atomic hydrogen was consistent with a source of free energy from the catalysis of atomic hydrogen by Sr^+ and Ar^+ .

Figure 3 The 656.3 nm Balmer α line width recorded with a high resolution visible spectrometer on the initial emission of a hydrogen-strontium rt-plasma and the emission at 70 hours of operation. Significant broadening was observed over time corresponding to an average hydrogen atom temperature of 20 eV



*H atom energy = 20 eV.

3.2 Balmer α line widths

The method of Videnovic et al. (1996) was used to calculate the energetic hydrogen atom energies from the width of the 656.3 nm Balmer α line emitted from rt-plasmas (Mills et al., 2002d, 2003c). The full half-width $\Delta\lambda_G$ of each Gaussian results from the Doppler ($\Delta\lambda_D$) and instrumental ($\Delta\lambda_I$) half-widths:

$$\Delta\lambda_G = \sqrt{\Delta\lambda_D^2 + \Delta\lambda_I^2}. \quad (5)$$

$\Delta\lambda_I$ in our experiments was 0.006 nm. The temperature was calculated from the Doppler half-width using the formula:

$$\Delta\lambda_D = 7.16 \times 10^{-7} \lambda_0 \left(\frac{T}{\mu} \right)^{1/2} \quad (6)$$

where λ_0 is the line wavelength, T is the temperature in K (1 eV = 11,605 K), and μ is the molecular weight (=1 for atomic hydrogen). In each case, the average Doppler half-width that was not appreciably changed with pressure varied by $\pm 5\%$ corresponding to an error in the energy of $\pm 10\%$.

The 656.3 nm Balmer α line widths recorded on the argon-hydrogen (90/10%)-strontium rt-plasma having a titanium filament initially and after 70 hours of operation are shown in Figure 3. Significant broadening was not observed initially. However, the Balmer α line profile of the plasma emission after 70 hours comprised two distinct Gaussian peaks, an inner, narrower peak corresponding to a slow component with an average hydrogen energy of 1 eV and an outer broader peak corresponding to a fast component of 20 eV. Only the hydrogen lines were broadened. These results are consistent with the catalysis of hydrogen to lower-states followed by subsequent transitions with increasing energy release by an autocatalytic mechanism previously reported with spectroscopic evidence (Mills and Ray, 2002a; Mills et al., 2002c).

We have assumed that Doppler broadening due to thermal motion was the dominant source to the extent that other sources may be neglected. This assumption was confirmed when each source was considered. In general, the experimental profile is a convolution of two Doppler profiles, an instrumental profile, the natural (lifetime) profile, Stark profiles, van der Waals profiles, a resonance profile, and fine structure. The contribution from each source was determined to be below the limit of detection (Mills et al., 2002b, 2002d, 2003a, 2003c).

The formation of fast H can be explained by a resonant energy transfer from hydrogen atoms to Sr^+ or Ar^+ ions of two and one times the potential energy of atomic hydrogen, respectively, followed by a collisional energy transfer to yield fast $\text{H}(n=1)$ as well as the emission of $q \cdot 13.6$ eV photons reported previously (Mills and Ray, 2002a; Mills et al., 2002c). For example, the exothermic chemical reaction of $\text{H} + \text{H}$ to form H_2 does not occur with the emission of a photon. Rather, the reaction requires a collision with a third body, M , to remove the bond energy- $\text{H} + \text{H} + M \rightarrow \text{H}_2 + M^*$ (Sidgwick, 1950). The third body distributes the energy from the exothermic reaction, and the end result is the H_2 molecule and an increase in the temperature of the system. In the case of the catalytic reaction with the formation of states given by equations (1) and (3), the temperature of H becomes very high.

3.3 Power balance of an Ar⁺ rt-plasma

Heat loss from the cell was primarily by radiation. Because the temperature of the kiln was fixed, the rate of heat loss from the cell, $P_T = P_{\text{loss}}$, was a function of the cell temperature:

$$P_T = f(T). \quad (7)$$

This relationship was determined from the argon-hydrogen control experiments in which $P = P_{\text{in}}$ was the electric power input. In the argon-hydrogen-strontium rt-plasma experiments, the rate of heat loss from the cell exceeded the electric power input by the excess power P_{ex}

$$P_T = P_{\text{in}} + P_{\text{ex}} = f(T). \quad (8)$$

The excess power was then

$$P_{\text{ex}} = P_T - P_{\text{in}} = f(T) - P_{\text{in}} \quad (9)$$

Using the measured cell temperature and the input power, the excess power of each argon-hydrogen-strontium rt-plasma was computed from equation (9).

Power input to the cell is plotted vs. the cell temperature for the argon-hydrogen-strontium rt-plasma and also for the argon-hydrogen control in Figure 4. The relation between cell temperature and the rate of heat loss from the cell, found from the argon-hydrogen control data, is of the form

$$P_T = f(T) = A(T^4 - T_0^4) \quad (10)$$

where $A = 2.74 \times 10^{-9} \text{ W/K}^4$ and $T_0 = 465^\circ\text{C} = 738.2 \text{ K}$ is the kiln temperature. Deviations of the control data from this expression are less than 2 W. Using equation (10) in equation (9) yields the excess power generated in the argon-hydrogen-strontium rt-plasma for each power input condition. Power input and excess power are tabulated in Table 1. The excess power ranged from 13.3 W at 110 W input to 26.0 W at 245 W input. The average excess power over the range was about 20 W. This corresponded to an excess power density of approximately 20 mW/cc in the one-litre cell.

The error in the temperature measurement over the range of 70°C was $\pm 0.5^\circ\text{C}$ for the type K thermocouples. However, the excess power was measured as the difference in filament power to the control to achieve the same temperature as the argon-hydrogen-strontium rt-plasma. Thus, the sources of error were the error in the calibration curve $\pm 2 \text{ W}$ and the power measurement of the watt meter ($\pm 1 \text{ W}$) in the power range of 100–300 W which was independent of the errors of the voltage ($\pm 0.25 \text{ V}$) and current ($\pm 10 \text{ mA}$) measurements due to any power factor. The propagated error of the calibration and power measurements was $\pm 2.2 \text{ W}$.

Figure 4 Plot of power input to the cell vs. the cell temperature for the argon-hydrogen-strontium plasma and also for the argon-hydrogen control. The calibration curve fit was $P_T = f(T) = A(T^4 - T_0^4)$ where $A = 2.74 \times 10^{-9} \text{ W/K}^4$ and $T_0 = 465^\circ\text{C} = 738.2 \text{ K}$ was the kiln temperature

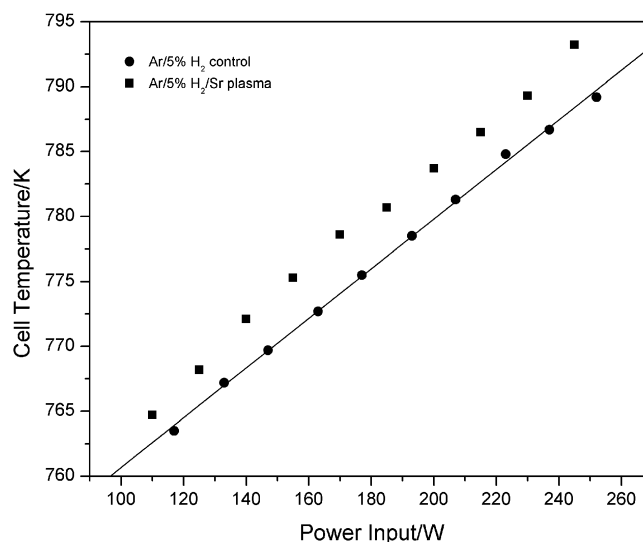


Table 1 Input and excess power for an argon-hydrogen-strontium rt-plasma

Voltage (V)	Current (A)	Input power (W)	Cell temp. (K)	Total power (W) ^a	Excess power ($\pm 2.2 \text{ W}$) ^b
40.4	2.73	110	764.7	123.3	13.3
41.7	3.04	125	768.2	140.6	15.6
43.0	3.46	140	772.1	160.1	20.1
43.4	3.57	155	775.3	176.3	21.3
44.4	3.85	170	778.6	193.3	23.3
45.7	4.27	185	780.7	204.2	19.2
46.2	4.48	200	783.7	219.9	19.9
46.8	4.70	215	786.5	234.8	19.8
47.7	4.83	230	789.3	249.8	19.8
48.4	5.17	245	793.2	271.0	26.0

^aEquation (10).

^bEquation (9).

3.4 NMR and FTIR of potassium chloro hydride KH^*Cl

Analyses consistent with the synthesis of KH^*Cl were given previously (Mills et al., 2000b, 2001a, 2001b, 2004). The ^1H MAS NMR spectra of six KH^*Cl samples from independent syntheses, the control comprising an equal molar mixture of KH and KCl, and the control KH relative to external tetramethylsilane (TMS) are shown in Figures 5(a)–(h), respectively. Ordinary hydride ion has a resonance at 1.1 ppm and

0.8 ppm in the KH/KCl mixture and in KH alone as shown in Figures 5(g) and (h), respectively. The sharp peak at 4.3 ppm and the broad peak at 6 ppm shown in Figure 5(g) are assigned to water in the KCl crystal and to KHCO_3 formed from air exposure of K during sample handling, respectively. The broad peak at 4.6 ppm shown in Figure 5(h) is assigned KOH formed from air exposure of KH during sample handling.

The presence of KCl does not shift the resonance of ordinary hydride as shown in Figure 5(g). The resonance at 1.1 ppm which is assigned to ordinary hydride ion was observed in the spectrum of each KH^*Cl sample as shown in Figures 5(a)–(f). The distinct 0.8 ppm and 1.1 ppm resonances could not be resolved if they were present. A large distinct upfield resonance was observed in each case at -4.4 ppm to -4.6 ppm which was not observed in either control. The upfield peak was stable for over one year and was the only novel peak in the range -300 ppm to $+300$ ppm as shown in Figure 6. The upfield peak is assigned to a novel hydride ion of KH^*Cl .

The experimental absolute resonance shift of TMS is -31.5 ppm relative to the proton's gyromagnetic frequency (Baldrige and Siegel, 1999; Mason, 1987). The KH experimental shift of $+1.3$ ppm relative to TMS corresponding to absolute resonance shift of -30.2 ppm matches very well the predicted shift of $\text{H}^-(1/1)$ of -30 ppm given by equation (4) wherein $p = 0$. The novel peak at -4.4 ppm relative to TMS corresponding to an absolute resonance shift of -35.9 ppm indicates that $p = 4$ in equation (4). $\text{H}^-(1/4)$ is the hydride ion predicted by using K as the catalyst (Mills and Ray, 2003b; Mills et al., 2004).

Figure 5 ^1H MAS NMR spectra of KH^*Cl samples and controls relative to external tetramethylsilane (TMS): (a)–(f) the spectra of six KH^*Cl samples from independent syntheses; (g) the spectrum of the control comprising an equal molar mixture of KH and KCl and (h) the spectrum of the control KH

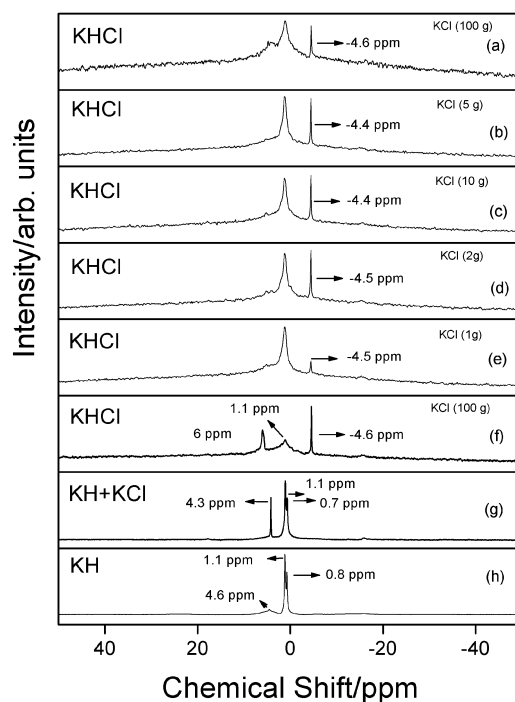
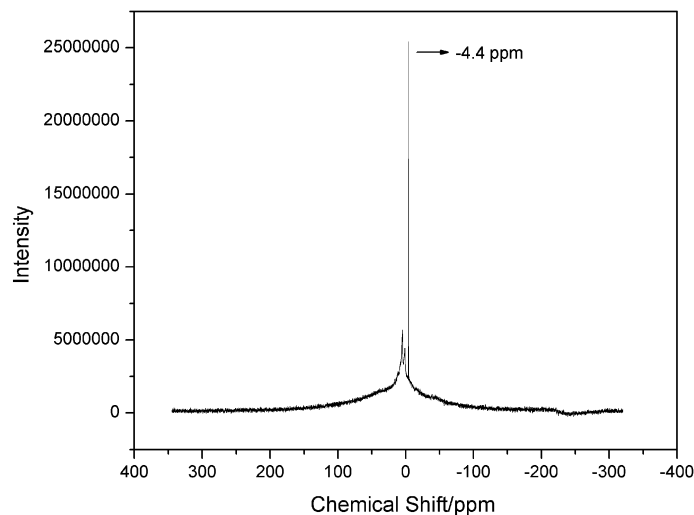


Figure 6 The ^1H MAS NMR spectrum relative to external tetramethylsilane (TMS) of a KH^*Cl sample that was stored under argon for one year before being acquired over the range -300 to $+300$ ppm

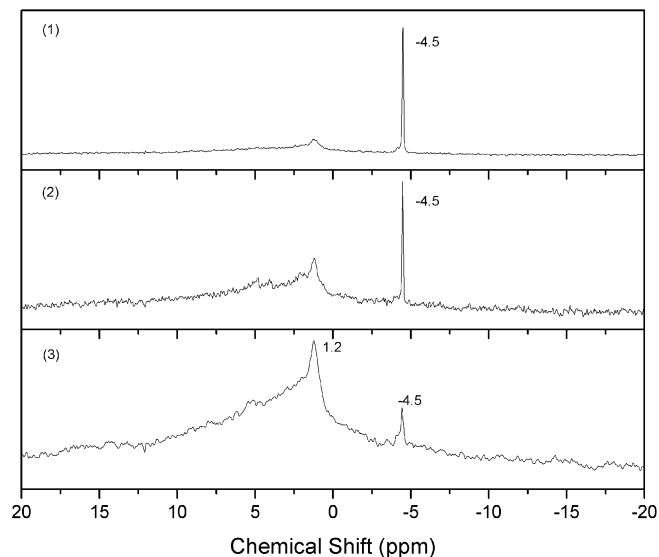
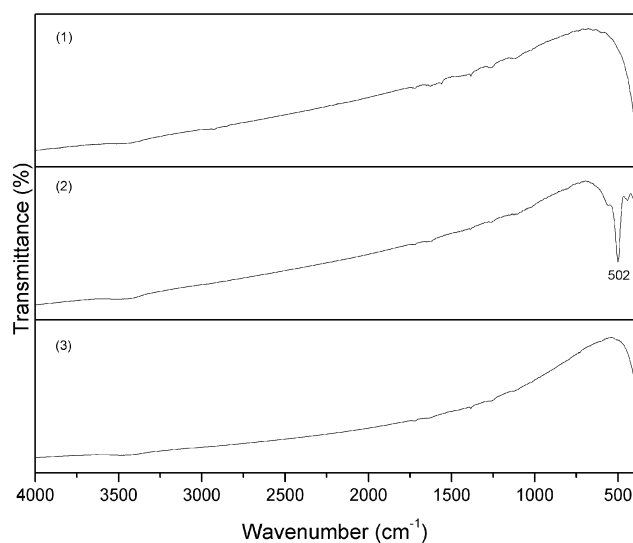
KH^*Cl samples exhibit different peak intensities at -4.5 ppm in the solid-state ^1H NMR spectra, likely due to a different H^* load in each sample. Samples with different intensities of the -4.5 ppm peak were analysed by FTIR spectroscopy to determine if there was any correlation between the -4.5 ppm ^1H NMR peak and possible IR vibration bands, in particular the substitutional H^- U centers band around 502 cm^{-1} . The list of the samples that were analysed by both solid-state ^1H NMR and FTIR techniques are given in Table 2.

Table 2 Summary of samples, and their ^1H NMR peaks and 'U'-H band (502 cm^{-1})

Sample ID	Content	^1H NMR peak (ppm)*	'U'-H band in IR (502 cm^{-1})
1	KH^*Cl	-4.5 (s), -4.14 (w), 1.2 (m)	No
2	KH^*Cl	-4.5 (m), 1.2 (m), 4.8 (w)	Strong
3	KH^*Cl	-4.5 (w), 1.2 (m)	No
4	KH^*Cl	-4.5 (s), -4.14 (m), 1.2 (w), 4.8 (m)	Strong
5	KH^*Cl	-4.5 (m), 1.2 (m)	No
Control	KCl	No -4.5 , 1.1 (w)	No

*s: strong; m: medium; w: weak.

A comparison of samples 1–3 having a relationship of decreasing -4.5 ppm ^1H NMR peak intensities is shown in Figure 7, and the corresponding IR spectra are shown in Figure 8. Sample 2 possesses the substitutional H^- U centers vibration band at 502 cm^{-1} ; whereas, samples 1 and 3 with a stronger and weaker 4.5 ppm ^1H NMR peak, respectively, do not. Thus, there is no relationship between the presence of U centres and the -4.5 ppm ^1H NMR peak.

Figure 7 Solid state ^1H NMR spectra of samples 1, 2 and 3**Figure 8** IR spectra from samples 1, 2 and 3

As further examples that support this conclusion, consider that samples 1 and 4 both show same strong intensity of -4.5 ppm peak in ^1H NMR spectra (Figure 9), but have markedly different FTIR spectra (Figure 10), in which sample 4 has the U H^- vibration band at 502 cm^{-1} , while sample 1 does not. The IR band at 3613 cm^{-1} and 3641 cm^{-1} in sample 4 could arise from interstitial H_2O , which corresponds to the 4.8 ppm peak in its ^1H NMR spectrum. Similarly, samples 2 and 5 both demonstrate medium intensity of -4.5 ppm peak in ^1H NMR spectra (Figure 11), but have different FTIR spectra (Figure 12), in which only sample 2 has the U H^- vibration band at 502 cm^{-1} .

Figure 9 Solid state ^1H NMR spectra of samples 1 and 4. Both samples have a strong -4.5 ppm ^1H NMR peak intensity

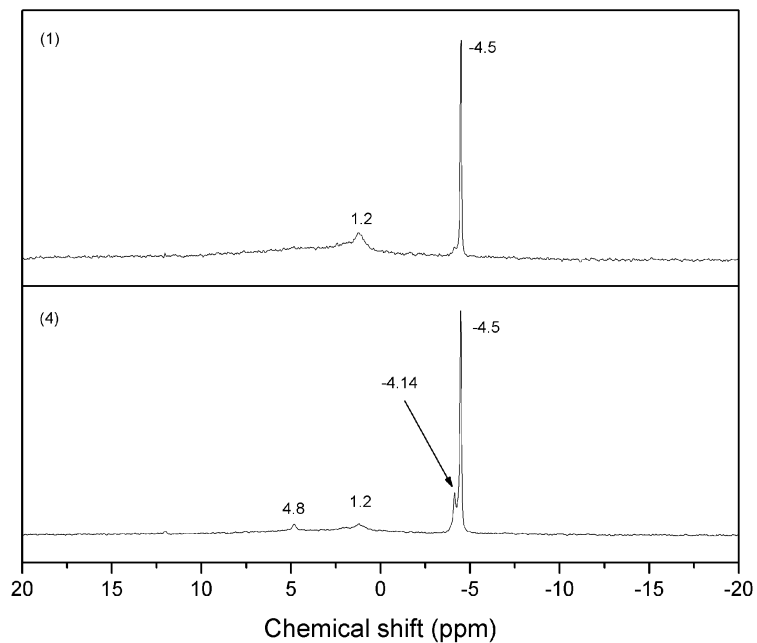


Figure 10 IR spectra of samples 1 and 4

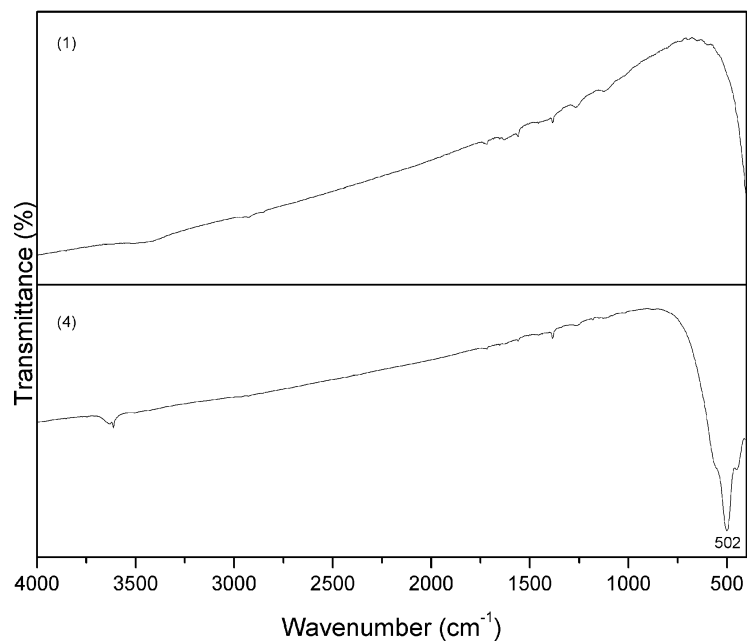
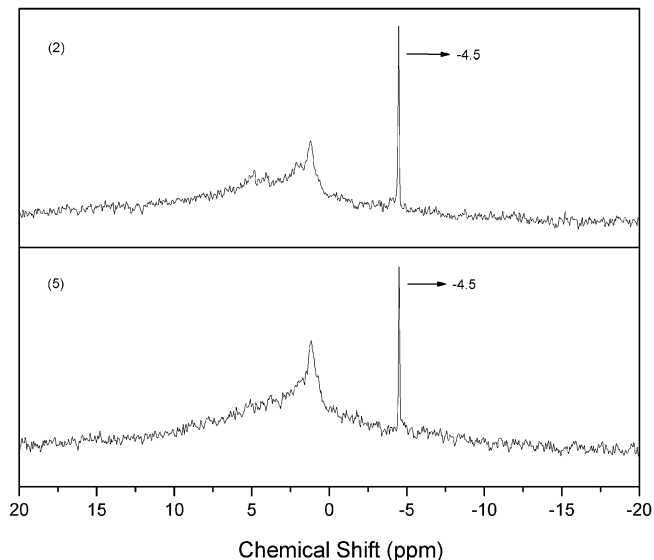


Figure 11 Solid state ^1H NMR spectra of samples 2 and 5. Both samples have a medium -4.5 ppm ^1H NMR peak intensity



In our IR study we did not observe a weak 850 cm^{-1} vibration band, assigned to the presence of interstitial ^1H ions (U1 centres) according to the literature. Other weak bands in the IR spectra of the samples are from KBr and KCl background (Figure 13). Since the NMR-FTIR comparison leads to the conclusion that the -4.5 ppm peak in ^1H NMR spectrum is not associated with the U H^- vibration band at 502 cm^{-1} bands, the -4.5 ppm peak in ^1H NMR spectrum is assigned to the $\text{H}^-(1/4)$ ion which matches theoretical predictions and is direct evidence of a lower-energy state hydride ion.

Figure 12 IR spectra of samples 2 and 5

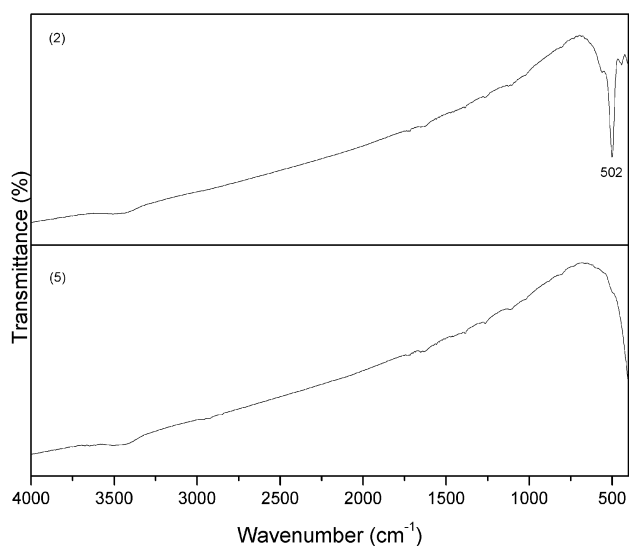
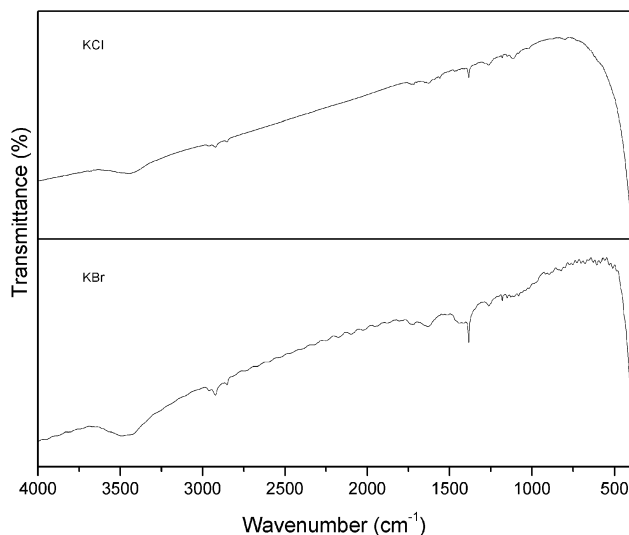


Figure 13 IR spectra of KCl and KBr

4 Summary

An rt-plasma formed with a low field (1 V/cm), at low temperatures (e.g., $\approx 10^3$ K), from argon and atomic hydrogen generated at a titanium filament with strontium which was vapourised by heating the metal. Strong Balmer emission was observed that indicated an energy source of >12 eV. The energetic reaction of atomic hydrogen was anticipated to form energetic hydrogen atoms. Significant Balmer α line broadening corresponding to an average hydrogen atom temperature of 20 eV was observed. The time-dependence of the appearance of fast H supported an energetic chemical reaction as the source. The power balance of a rt-plasma formed with Ar^+ as the catalyst was measured by heat loss calorimetry. The steady state temperature of a rt-plasma formed with strontium and increased by Ar^+ was significantly higher than heated argon which did not form a rt-plasma. A maximum excess power of 26 W was observed. The enthalpy of formation ΔH_p of strontium hydride is -47.59 kcal/mole (1.0 eV/H atom) (Mueller et al., 1968). Thus, the energy for hydriding all of the 1.2 g (13 mmoles) of strontium would be 0.65 kcal compared to the energy released over the minimum three hours to steady state of 280 kcal. Thus, the excess power of $20 \text{ mW} \cdot \text{cm}^{-3}$ measured calorimetrically on rt-plasmas formed by Ar^+ as the catalyst supported the rt-plasmas mechanism since there is no known chemistry which could account for the observed power.

Substantial evidence of an energetic catalytic reaction of rt-plasmas of intense hydrogen Lyman emission, a stationary inverted Lyman population, excessive afterglow duration, highly energetic hydrogen atoms, characteristic alkali-ion emission due to catalysis, predicted novel spectral lines, and the measurement of a power beyond any conventional chemistry was previously reported (Mills et al., 2004) involving a resonant energy transfer between hydrogen atoms and K to form very stable novel hydride ions $\text{H}^-(1/p)$ called hydrino hydrides having a predicted fractional principal quantum number $p = 4$. Since the comparison of theory and experimental shifts of KH^*Cl is direct

evidence of lower-energy hydrogen with an implicit large exotherm during its formation, the NMR result were repeated with the further analysis by Infrared spectroscopy (FTIR). The ^1H MAS NMR spectrum of novel compound KH^*C relative to external tetramethylsilane (TMS) showed a large distinct upfield resonance at -4.4 ppm corresponding to an absolute resonance shift of -35.9 ppm that matched the theoretical prediction of $p = 4$. The FTIR results eliminated any known explanation such as U centred H for the assignment of the extraordinary upfield-shifted NMR peak. The spectroscopic, chemical, and thermal results show the possibility that a novel reaction of atomic hydrogen that uses certain catalysts such as Sr^+ , Ar^+ , and K to form more stable hydrides may be a clean new energy source.

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