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A SLIDING-SHAFT VACUUM LOCK FOR A MASS SPECTROMETER*

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INTRODUCTION

The sliding-shaft vacuum-lock type of sample changing mechanism for a mass spectrometer permits full utilization of instrument time. With it a solid sample can be changed in the order of a minute without degrading the vacuum in the spectrometer. Although now used extensively and reliably, early models of the vacuum lock were fraught with shortcomings. A history of its development will be presented here. A new version will also be described in which the sample carriage and differentially-pumped manifold are made entirely of sintered alumina (96 percent Al_2O_3).

HISTORY OF SLIDING-SHAFT VACUUM LOCK DEVELOPMENT

The sliding-shaft vacuum lock had its origin in the O-ring sealed rods used as motion feedthroughs in vacuum systems. From this beginning engineers at the Kellex Corporation¹ conceived of a system for introducing solid samples into a mass spectrometer without having to vent the source region to the atmosphere (Fig. 1-a). A rod carrying the sample was inserted into the source region through an O-ring seal followed by three pumping chambers having successively lower pressures. The pumping chambers were separated by simple bellows-sealed stoppers which were held shut by atmospheric pressure and had to be toggled open when the rod was inserted. The sample traversed the pressure region from atmospheric to that of the analyzer tube without degrading the high vacuum in the tube. The axis of the sample carriage coincided with the axis of the analyzer.

This device was followed by an improved model² in which O-ring seals were provided between each of the pumping chambers and the stoppers were

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replaced by spring-loaded flap valves (Fig. 1-b). The device was also repositioned so that its axis was perpendicular to the analyzer tube axis.

Further development³ eliminated the flap valves (Fig. 1-c). A dummy carriage was used to plug the openings between pumping stages upon extraction of the sample carriage. The two carriages were joined together by a bayonet connector. With a sample in place, the dummy was housed in a receptacle opposite the pumping manifold. With the sample carriage removed, a catch held the dummy out against atmospheric pressure.

This arrangement had a serious disadvantage⁴ in that the dummy could be disengaged and lost in the vacuum system if the sample carriage were rotated 1/4 turn during insertion or removal. If the dummy were lost while the carriage was not in place, it would be shot violently into its receptacle allowing a sudden complete loss of vacuum throughout the system.

The sample carriage was made of Mykroy, a semi-sintered vitreous material which could be machined. This material was poorly suited to the application because of its brittleness and porosity.

Electrical connections to the filament were made through sliding contacts in the sample carriage which mated with spring-loaded contacts in the source structure. The carriage contacts became coated with grease from sliding past the O-rings resulting in uncertain electrical contact. Furthermore, heat from the sample filament was conducted to the grease on the contacts causing it to outgas copiously. The pressure rise and resultant loss of resolution forced termination of an analysis after only a short run.

A much more practicable vacuum lock was described by Stevens⁵ in 1953 (Fig. 2-a). It had differentially-pumped manifolds on each side of the source block. With this double-ended design, atmospheric pressure was

always balanced. There was no hazard of losing dummy carriages in the vacuum system. The vacuum lock was also double-acting with two sample carriages being used alternately. As one carriage was removed, the other was simultaneously inserted from the opposite end and the unattached dummy was shuttled back and forth between them.

O-ring seals were provided only at the atmospheric end of the manifolds to reduce leakage into the first pumping stage. Leakage between successive stages was kept low by small radial clearances (0.002- to 0.004-in.) between the carriages and manifolds. Since the carriages did not ride on O-rings their entire length, it was necessary for them and the manifolds to have good bearing surfaces. The carriages and manifolds of Stevens' vacuum lock were made of stainless steel which galls readily. To prevent galling the parts were lubricated with Aquadag.

Stevens' vacuum lock did not suffer from heavy outgassing upon heating of the sample filament. The filament and source parts which he mounted on the sample carriage were recessed so that they remained clean in passage through the lock. Electrical leads were brought to these source components through the hollow sample carriage and a vacuum-tight glass seal.

The use of stainless steel against stainless steel proved to be a mistake. In a later version,⁶ Stevens and Inghram chose bearing surfaces of different metals. Stainless steel was still used, but the manifold was case-hardened by Malcomizing - a nitriding process - and the carriage was hard-chrome plated. These very hard surfaces (Malcomized stainless steel: about 1000 Brinell, chromium: 850-900 Knoop) were operated without any lubricant. No O-rings were used either. Leakage from the atmosphere and between stages was reduced by providing a radial clearance of only 0.0005 inch between the carriages and manifolds.

Several variations of Stevens' design have since appeared. Echo and Morgan⁷ found that a considerable saving in pumpdown time resulted from the use of only one carriage for running samples. The carriage which was exposed to the atmosphere during a run apparently outgassed when used again. Consequently, they made the dummy long enough to fill both manifolds when the single carriage was removed for sample changing (Fig. 2-b). Since they had experienced scoring of the Malcomized surface, they plated both contacting surfaces of the vacuum lock with hard chrome.

A copy of Echo and Morgan's vacuum lock was constructed in our laboratory several years ago. Our experience has shown that even the hard chrome surfaces are susceptible to seizing if abrasive material finds its way between the carriage and manifold. One of our routine operating procedures is to clean the carriages frequently with acetone to prevent such an occurrence. However, without a lubricant chromium against chromium does wear leading to an eventual reappearance of the problem. After approximately 4000 sample changing cycles in five years of operation, the chrome plate on our vacuum lock wore through or thin enough in spots for scoring and seizing to occur. This observed lifetime is undoubtedly a function of the quality and thickness of the chromium layer and the technique of the person who operates the lock. Wear can also be hastened by a very slight misalignment of the bores in the two manifolds.

An inherent problem in the double-ended design is the critical alignment required between the two pumping manifolds to permit free passage of the carriages with a clearance of only 0.0005 inch. Boerboom, et al.,⁸ designed a double-ended and double-acting vacuum lock which alleviated this problem. They made the sample and dummy carriages in five pieces and connected them together flexibly. In this way strict alignment of the bores

in the two manifolds was not essential. However, the fabrication was quite complex. Another model they built had a long one-piece shaft which served as both sample carriage and dummy (Fig. 2-c). The lock was double-ended but single-acting. In operation the carriage would be extracted from one end just far enough to permit changing of the filament assembly.

In the vacuum lock of Nuclide Analysis Associates, the alignment problem is remedied by not making the manifolds demountable. The manifolds and source block are welded into a single unit. The bore of both manifolds can then be ground and polished in one operation.

W. E. Duffy⁹ avoided the alignment problem by reverting to Kellex Corporation's single-ended design. With a pumping manifold on one side of the source block and a receptacle for the dummy on the opposite side, no alignment of bores is required. Duffy joined the sample and dummy carriages together so there was no possibility of the latter being lost in the vacuum system. Through the simple expedient of a counterweight to balance atmospheric pressure, the lock could be operated with as little effort as a double-ended lock.

THE ALUMINA VACUUM LOCK

An increased sample load in our laboratory made it necessary to equip our second mass spectrometer with a rapid sample-changing system. Difficulties experienced with the chrome-plated vacuum lock in use dissuaded us from duplicating it. From an investigation of existing designs, it appeared that none presented a completely satisfactory solution to the problem presented by two close-fitting surfaces sliding against each other with no lubrication. Our subsequent work resulted in a sliding-shaft vacuum lock which effectively solves this problem.

Our design is presented schematically in Fig. 3. We have incorporated

features of several previous designs with refinements of our own. A one-piece shaft, 1 1/4 inches O.D., serves as sample carriage and dummy. The lock itself is single-ended. We believe this has distinct advantages over the double-ended design. Foremost is the elimination of the alignment problem. In addition, with all other things equal, only one-half of the pumping capacity is required at each stage to achieve a given pressure. A simple vacuum envelope to house the dummy end of the shaft is also easier and cheaper to fabricate than another pumping manifold although this is offset by the necessity of providing a counterweight.

The counterweight is such that one or two pounds of unbalanced atmospheric pressure hold the carriage firmly in the source. A latch holds the carriage in its retracted position. If the carriage is inadvertently unlatched, friction in the counterweight pulleys prevent the carriage from moving into the source.

If it is desirable to completely remove the sample carriage and attached source plates from the mass spectrometer for cleaning, checking alignment, etc., one must use a two-manifold system. If not, a single-ended system seems preferable for the reasons stated.

Our solution to the problem of seizing is not evident from the diagram. It consists in the material used for bearing surfaces. In our vacuum lock the manifold and carriage are made entirely of high-strength alumina - 96 percent Al_2O_3 . The property of this material which makes it so attractive as a dry bearing is its extreme hardness (see Table). Sintered alumina has a hardness of 2100 on the Knoop scale which is more than twice the hardness of chromium. Tests we have made lead us to believe that it is virtually impossible for this vacuum lock to score or seize. We have poured powders of metal, sand, alumina, and diamond into

the annulus between the carriage and manifold and then worked the carriage back and forth. The foreign material introduced, depending on its hardness, is either pulverized or laps the vacuum lock surfaces together.

TYPICAL PROPERTIES OF HIGH-STRENGTH ALUMINA

Composition	95% Al_2O_3
Porosity	Vacuum tight (by helium mass spectrometer leak detector)
Water absorption	0.00%
Specific gravity	3.6
Hardness	2100 Knoop
Tensile strength (25°C)	25,000 psi
Compressive strength (25°C)	300,000 psi
Working temperature (max.)	1600°C
Coefficient of thermal expansion (25-600°C)	8×10^{-6} per °C
Dielectric strength (25°C, 60 cycle A.C.)	230 volts per mil

The fact should also be mentioned that alumina can be ground to close dimensional tolerances and high surface finishes. Our carriage and manifold fit together with a clearance of 0.0005 inch and have a surface finish of 12 microinches rms.

Another advantageous property of alumina for this application is its high dielectric strength. The first plate of the ion lens is mounted directly on the sample carriage without the need of intermediate insulators. The specially designed filament hat (Fig. 4) fits into a central cavity in the sample carriage where it is isolated from the lens plate and from ground. Material vaporized from the sample filament could conceivably form a conductive layer on the surface of the sample carriage. However, two

circumferential grooves, 1/8-in. wide by 1/8-in. deep, on the carriage prevent the formation of a leakage path to ground.

High voltage leads for the filament and source parts on the sample carriage have generally been fed through the end of the carriage which is handled by the operator. Although shields and interlocks can be provided to protect personnel, it seemed preferable to remove the hazard to a more isolated location. This has been accomplished in our design by feeding the necessary voltages and current (routinely 12 KV ion-accelerating voltage and 3.5 amperes filament current) into the source region through a ring of ceramic-insulated bushings mounted around the dummy housing. From there, connections are made to the filament and first lens plate on the sample carriage by means of a bellows-sealed linear motion feedthrough. Three contact buttons on the motion feedthrough are individually spring mounted so that reliable contact is maintained even during slight rotational adjustments of the sample carriage.

Although the central section of the sample carriage does become heated during an analysis, the resulting thermal expansion is not sufficient to cause binding. In a test the sample carriage could be retracted immediately after continuous heating of a sample filament at 1600°C for three hours. In spite of the heating of the carriage, no abnormal outgassing has been observed other than that normally attributed to the sample filament itself.

CONCLUSION

An alumina vacuum lock has been described which effectively eliminates seizing of the sliding surfaces. What might be considered a disadvantage with alumina parts is that they cannot be fabricated in the average machine shop, even one extensively equipped. Parts must be procured to specifications from vendors specializing in ceramic materials. The cost is several

times greater than the cost of the equivalent item constructed of chrome-plated stainless steel. However, in our own laboratory the analytical time lost in restoring the chrome-plated lock to service has far exceeded in value this initial cost differential.

ACKNOWLEDGMENT

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(a) First version (Ref. 1)

(b) Second version (Ref. 2)

(c) Final version (Ref. 3)

FIG. 1. KELLEX CORP. VACUUM LOCKS

(a) Double-ended, Double-acting (Stevens, Ref. 5)

(b) Double-ended, Single-acting, Separate sample carriage and dummy (Echo and Morgan, Ref. 7)

(c) Double-ended, Single-acting, Integral sample carriage and dummy (Boerboom, et al., Ref. 8)

FIG. 2. SLIDING SHAFT VACUUM LOCKS

FIG. 3. ALUMINA VACUUM LOCK, SINGLE-ENDED

FIG. 4. FILAMENT HAT FOR ALUMINA VACUUM LOCK