Paper Number 2006-01-0379

Simplifying the Approach to Specify and Measure Product Seal Integrity and Leak Tightness

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ABSTRACT

A common problem encountered when designing a new product for demanding environmental conditions is specifying its leak tightness and measuring its seal integrity. Correlating empirical test data to production leak testing is an on-going challenge and a major source of confusion. Products of interest are those in automotive fuel and vapor management systems, A/C and cooling systems and power-train components and systems.

A simple solution to the problem is proposed in this paper, based on defining the maximum acceptable microchannel geometry called the Equivalent Micro Geometry (EMG). Various mechanisms of fluid transport through microchannels are summarized. Flow through two types of EMGs, sharp edged orifices and microchannels, are examined. A practical implementation of the EMG approach to determine leak tightness specifications of components, exposed to automatic transmission fluid, is presented.

Newly developed Intelligent Micro Flow Sensors enable, for the first time, direct measurement of airflow through microchannels in the viscous flow, slip flow, transitional and molecular flow regimes. This technology is used to demonstrate the implementation of the EMG concept for setting up an inline production leak tightness test with air, to provide cost effective solutions.

INTRODUCTION

Several automotive disciplines are experiencing increasing demands to maintain high levels of leak tightness and seal integrity, while reducing product manufacturing and warranty costs. The automotive fuel systems and evaporative management systems are required to comply with hydrocarbon emission standards (US Tier 1 or 2 Evap., Carb Lev-I or II Evap. or Euro Stage 3, 4 or 5 Evap). The air conditioning and cooling systems have to meet EPA refrigeration emission standards. Power-Train applications are required to reduce warranty costs and enhance safety.

The total permissible emissions for fuels, hydrocarbon vapors, oil coolants and refrigerants are typically defined by mass flow rates (mg/day, mg/year etc.) of the specific fluid. The emission is a result of diffusive permeation and fluid leakage. There is an increasing requirement for elimination or reduction of emissions due to leakage. Therefore, product engineers, process engineers and quality engineers need to find a way to translate the requirements into production specifications, where, typically, air or tracer gases (like helium) are used for leak testing.

Conventionally, production leak tightness specifications have been set in terms of the maximum allowed gas flow (air or tracer gases such as helium) at test conditions, for a given gas. The drawback of this method is its dependence on the leak test method used, thus creating difficulties in correlations and causing confusion in users not familiar with flow terminology.

There is a long history of attempts to find analytical tools to correlate a product's operational leak tightness requirements with its production leak testing specifications. The Hagen-Poiseuille equation was applied to correlate pure isobutene to helium flow rates through capillary channels (1). According to this viscous flow model, flow rate through circular channels, for low Reynolds number flows, is calculated using

$$Q = C\frac{d^4 \Delta P}{L\mu} \tag{1}$$

where Q is the volumetric flow rate, d is the diameter of the channel, ΔP is the pressure differential, L is the length of the channel, μ is the dynamic viscosity of the fluid and C=$\pi/128$ or a constant defined during calibration. However, as the author clearly states, correlations based on this model are limited to a narrow, low differential pressure range. Moreover, this model is based on the no-slip condition, which is not applicable when dealing with channels of a few microns. For a given channel geometry and pressure differential (3,4), slip phenomenon causes flow rates to be higher than what equation (1) suggests, while 'choking' in compressible fluids causes flow rate to be significantly lower.

Models for calculating gas flow through stainless steel crimped metal capillaries, using slip flow corrections, offer better gas flow correlations and yield better accuracy (3,7). Those models are limited to pure gases, low pressure-differentials and are applicable for specific flow regimes and conditions.

Production leak tests of many fuel components are done at conditions that do not meet these assumptions. The existing ASTM standard (2) offers analytical correlation between the operational fluid and test gas (helium, air) flow using the same model, while suggesting a safety factor of 10 or more to any such calculation.

Other numerical solutions have been suggested for compressible and non-compressible fluids (3,4,7). Many of the existing models (3,7) for gas flow and gas-vapor mixture flow are based on molecular considerations.

For gas microflows the Knudsen number, *Kn* (ratio of mean free path of gas molecules to the characteristic length of the microchannel) is used as an indicator of the flow regime. Kn < 0.01 represents continuum flow with negligible slip effects and Kn> 10 represents free molecular flow (7). For inert gas flow, a generic model, based on gas flow through a microchannel at any Kn and free molecular flow for low pressure differential (non-choking conditions), was suggested by Knudsen (and Kennard).

$$\frac{\dot{m}_{Kn}}{\dot{m}_{fm}} = \frac{3\pi}{64\overline{Kn}} + \frac{1+2.507\overline{Kn}^{-1}}{1+3.095\overline{Kn}^{-1}} \qquad (2)$$

$$\dot{m}_{fm} = \frac{\pi d^3}{\sqrt{2RT}} * \frac{\Delta P}{L} \qquad (3)$$

where $\dot{m}_{Kn}$ is the mass flow at any arbitrary Knudsen number, $\dot{m}_{fm}$ is the mass flow rate in the free molecular flow regime, and $\overline{Kn}$ is the Knudsen number averaged along the flow direction. This model gave better results, compared to equation (1), when applied to inert gas or vapor flow through microchannels

Liquids (or vapors) participating in real life leakages cannot be assumed to be 'pure substances'. Understanding the actual operational microscale fluid transfer mechanisms is critical to establish realistic leak tightness specifications.

Slip effects (4), surface tension effects (5), capillary forces (6) and micro-electrostatic boundary layers (7) are some of the prominent phenomena observed at micro scale. Slip effects come to play when *Kn* > 0.001, and cause flow rates to be higher than those predicted by equation (1).

Surface tension effects are expressed by the classical LaPlace equation (5).

$$P = 4\gamma Cos\Theta / d \qquad (4)$$

where P is the driving gage pressure at the microchannel outlet, γ is surface tension and Θ is the contact angle between the surface and the microchannel wall. Smaller the microchannel diameter, larger the gage pressure required to 'break' the surface tension and cause leakage. A liquid with higher surface tension is expected to leak less.

Capillary electrostatic forces result from the electrochemical attraction between microchannel walls and the liquid. Higher the capillary electrostatic forces, fewer the liquid drops that leave the surface.

Micro electrostatic boundary layers are formed due to the flow of a polar liquid through a microchannel. When a polar liquid flows through a microchannel, the surfaces of the channel walls acquire an electric charge, which then influence the migration of charges within the liquid. A boundary layer of fluid ions, known as the Stern layer (7), adheres strongly to the channel wall. This layer influences the liquid and causes a thicker layer of excess charges called the Diffuse or Gouy-Chapman layer. The two layers, combined, are called the Electric Double Layer (7) (EDL). The effect of these layers is to reduce the effective microchannel diameter that can transport the liquid, and an increase in the drag forces opposing the flow, thereby reducing or inhibiting leakage. The EDL thickness is a strong function of the fluid and wall electrical properties (Zeta potential, dielectric constants, wall electric potential) and temperature (7). This phenomenon reduces leaks through channels of diameters 2-μm and less.

Real life fluids include contaminants and additives, which may be as large as few microns. For instance, refrigeration fluids contain refrigerants (such as R134A) and compressor lubricants (typically polyalkylene glycol with additives). Gasoline (8) and diesel also contain additives. Such additives can cause a microchannel to get plugged after a relatively short period of time. Furthermore, as the leaking fluid is exposed to atmospheric conditions, evaporation of liquid components with lower vapor pressure takes place, generating a skin of non-homogeneous fluid. The effect of liquid non-homogeneity and contamination is to reduce leak rates by 'plugging' the leak paths. A mathematical expression of this "plugging" effect does not exist, but can be assumed to be a function of the fluid type, particulate sizes, fluid viscosity, microchannel diameter, microchannel length and the driving pressure.

In-lieu of the complexity involved in calculating a realistic correlation between operational fluid leak tightness requirement and production leak tightness specification, an alternative method is offered. This method is based on defining the maximum allowed leak geometry, also called the Equivalent Micro Geometry (EMG). This method, based on empirical tests, is quite universal for specific automotive systems (fuel handling and delivery, high pressure automatic transmission fluid applications etc.) and is simple to use and correlate between various production leak technologies and test stations. It has recently been used as the SAE Standard (8) for Fuel System Component leak test specifications.

The application of the EMG concept, for determining production leak test specifications for a sample automatic transmission fluid, is presented. Also presented is a unique air micro-flow measurement technology (9,10,11) for leak measurement that acts an alternative to helium mass spectrometry, and therefore, meets automotive requirements to simplify the manufacturing and test processes and to reduce equipment and operation costs.

DEFINITIONS AND APPROACH

The Equivalent Micro Geometry (EMG) is a controlled and reproducible largest micro-geometry that will allow no liquid or vapor leak, for given operating conditions and fluids. Since the purpose of production leak testing is to find leaks through one or more ‘pin holes’ or micro-geometries, it is suggested that the product leak tightness specification be expressed as the maximum allowed micro-geometry or the Equivalent Micro Geometry (EMG).

Leak paths may exist as sharp-edged orifices, narrow microchannels or have some random shape. Sharp edged orifice leaks can be simulated using stainless steel discs with laser-drilled microholes, also referred to as Equivalent Diameters (EDs). They cause the maximum amount of leak possible through a leak path of a particular diameter. They have a small length to diameter ratio (L/d < 10). Narrow microchannels can be simulated by finely controlled silica microchannels, also called Equivalent Channels (ECs). They have a small length to diameter ratio (L/d > 100). They represent leak path through a straight and narrow channel through the entire thickness of the wall material. For a particular diameter, leak through an EC will be less than an ED.

Equivalent Micro Geometry (EMG) is a generic term for Equivalent Channels (ECs) or Equivalent Diameters (EDs). As most defects have some associated length (due to the wall thickness), it is desirable to use ECs more than EDs. Furthermore, existing microfabrication technologies enable us to produce such controlled microchannels in a repeatable fashion. The diameters of the microchannels can be held with a variation of less than ±0.5 µm. Figure 1 presents images of the inlet and outlet cross-sections of a 15-µm EC.

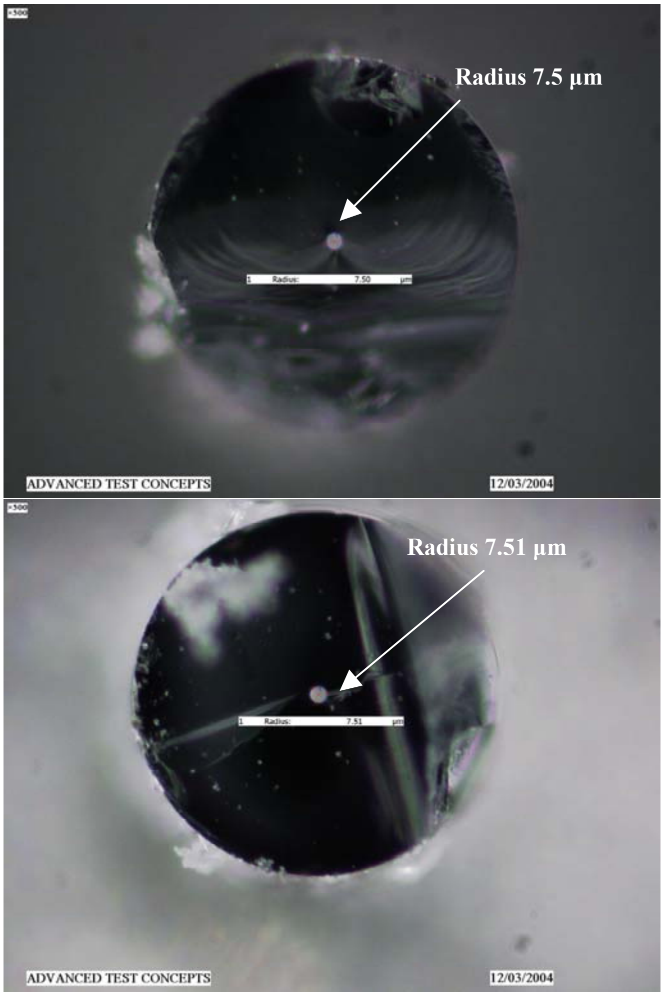

Figure 1: Images of EC inlet (top figure) and outlet (bottom figure) diameter with magnification 500x. The EC has nominal radius of 7.5-µm and a nominal length of 3.5 mm. Note the consistency in the inlet and outlet geometries.

As there are significant differences between ED and EC flow properties, one must clearly specify the selected EMG. Due to the complexity of the leakage mechanism, the maximum allowed EC or ED can be determined by conducting a controlled experiment. The experiment needs to be conducted for particular fluid at its maximum operating conditions, not for every product. For instance, testing with automatic transmission fluid (ATF) at the maximum operating pressure and temperature will cover all transmission components and cavities exposed to such conditions.

Once the Equivalent Channel or Equivalent Diameter is defined by the experiment, it becomes the Leak Tightness Specification. This Leak Tightness Specification, unlike many existing ones, is not dependent on the specifics of parts design, construction or manufacturing process or test method.

Real life defects have random, inconsistent geometries and are not perfect EDs or ECs. Therefore, the question of what should be used as the EMG is legitimate, and is examined in the following sections.

The purpose of the production in-line or sampling leak testing is simply to assure that seal tightness of each manufactured product is better than the EC/ED, as will be explained in the following sections. Another advantage of this method is that the method of leak testing (micro-flow, tracer-gas, pressure decay, etc.), test parameters or measurement units assume secondary importance, since each leak test is based on comparison to the EMG leak performance at those test conditions.

TEST PROCEDURE

The process of specifying Product Seal Integrity and Leak Tightness using the concept of Equivalent Micro Geometry must start with a controlled application test, an example of which is described here.

The concept was used to establish the EMG through which automatic transmission fluid (ATF), Dexron®[1] VI, will not leak. The test results can be used to define the seal integrity of components, castings, weldings and seals that will contain Dexron® VI at high pressure and temperature. A gravimetric test was setup, in which different sizes of EMGs were subjected to Dexron® VI at a pressure of 300-psi and a temperature of 300 ºF. The ATF leak was collected in collection nuts and the increase in collection nuts' weight was measured at regular intervals. The test was carried out for duration of 3 days and measurements were taken at the 24-hr, 48-hr and 72-hr intervals.

Equivalent Channels were fabricated by bonding circular silica microchannels, of a predetermined length, to the center of stainless steel discs while Equivalent Diameters were fabricated by laser-drilling micro-holes in stainless steel discs. Both were housed in Swagelok® VCR ¼" stainless steel holders, also referred to as EMG holders. ECs with nominal length of 3.5 mm and the following diameters were used: three of 10 µm, three of 15 µm, three of 20 µm, three of 25 µm and three of 30 µm. EDs with the following nominal diameters were chosen: two of 5 µm, two of 7 µm, two of 10 µm, two of 15 µm, three of 19 µm and two of 22µm. Four blank discs, without any holes, were added to the ECs and EDs. The purpose of the using blank discs with the samples is to provide a reference point against which leaks through EMGs will be evaluated.

The diameters of all EMGs were gauged using ATC's Gauging Microscope (Keyence Model No. VH8000, SN: 2020720). It was observed that the ECs have very finely controlled geometries (presented in figure 1). EDs, with diameters less than 20 µm, were found to have somewhat rough edges.

The flow rate of nitrogen through each EMG was measured, at inlet pressures ranging from 10-psig to 150-psig. The measurements were made using ATC's primary PVTt standards (RD09 and RD09A) and ATC's Microflow Sensor Standard (PN: IF-010C-100S). The purpose of nitrogen flow measurements is to compare before-test flow readings and after-test readings and determine if the EMGs got clogged during the test.

The EMG holders were carefully filled with Dexron® VI, purged of air and installed on a test fixture, as shown in figure 2. The test fixture was built to have tube connections from a Dexron® VI reservoir (not shown in the figure) to 18 EMG holders simultaneously. Collection nuts, with absorbent material, were weighed on a balance (Mettler Model AE240), and loosely hand-tightened on the bottom of each EMG holder. Aluminum foil cups were placed under the EMG holders to act as 'overflow catchers' in case there is excess leak from any EMG. The manifold was filled with Dexron® VI from the reservoir ensuring that there are no air bubbles in the liquid. Station valves (manual plug valves) were used to isolate EMG holders from the Dexron® VI manifold, in case of an excess leak.

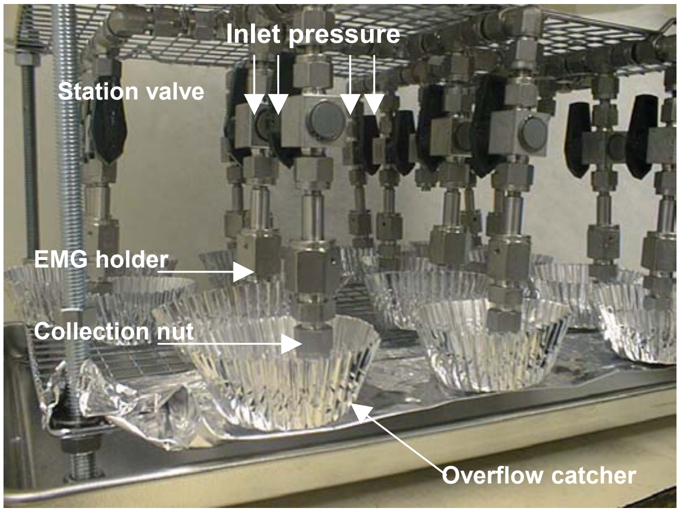

Figure 2: Setup of ATF test manifold. Leakage of Dexron® VI, at 300 psia and 300 ºF, through Equivalent Channels and Equivalent Diameters of different sizes, was measured by periodically measuring the increase in weight of collection nut.

The test manifold was placed in an environmental chamber (Tenney Model number BTS S/N 26024-10). The temperature in the chamber was increased to 300 ºF and test pressure of 300 psia was applied on top of Dexron® VI in the reservoir using a precision venting regulator. The environmental temperature and test liquid pressure were monitored using ATC's temperature sensor (RTD Pt100) and a silicon micromachined pressure transducer respectively.

The EMGs were checked for gross leaks and, in case of leakage into overflow catchers, the corresponding station valves were closed. The collection nuts were weighed at the 24-hr, 48-hr and 72-hr periods. The

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[1] Dexron® is a registered trademark of General Motors Corporation.

collection nuts were handled with powder-free latex gloves to minimize contamination.

At the end of the test, a cotton swab was used to absorb remnant Dexron® VI that had collected downstream of each EMG but not flowed into the collection nut. The increase in weight of the cotton swab was added to the total increase in weight of the corresponding collection nut.

The EMG holders were uninstalled from the manifold and drained of Dexron® VI. The EMGs were flow tested with nitrogen using ATC's microflow standard, at inlet pressures varying from 10-psig to 150-psig. The flow values were compared with the flow values at the beginning of the test.

OIL THROUGH EMG TEST RESULTS AND DISCUSSION

For an EC and ED of the same diameter, gas flow rate through the ED is more than that through the EC. This is primarily because of higher differential pressure per unit length and lesser wall friction. This is shown graphically in figure 3, where nitrogen flow rate though ECs and EDs at 15-psig is compared. The measurement was made using ATC's microflow standards with accuracy better than +/-1% of reading.

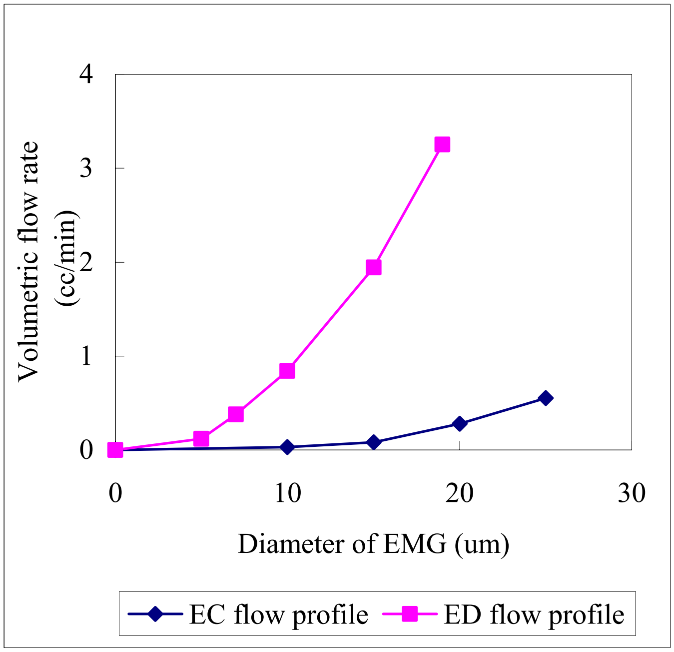

Figure 3: Nitrogen flow rate though ECs and EDs. For the same diameter, flow through an ED is more than an EC.

Uncertainty in measurement of weight was determined by analyzing the variability in weight of collection nuts installed on the blank disc holders, since there was no oil leak through them. The variability is attributed to operator handling inconsistency, equipment repeatability errors, absorbent material loss, oil evaporation and accumulation of minute dust particles on collection nuts. The total measurement uncertainty over the period of 3-days was determined to be ± 0.17 mg/day.

The total leak rate of Dexron® VI through EMGs of various sizes at 300 ºF and 300 psig is presented in figure 4. It is seen that leak rate is a strong function of the EMG diameter. The maximum measurable leak with the experimental setup was approximately 190 mg of Dexron® VI. Any further leak was beyond the capacity of the collection nuts and dripped into the overflow catchers. In such a case, the corresponding station valve was promptly closed.

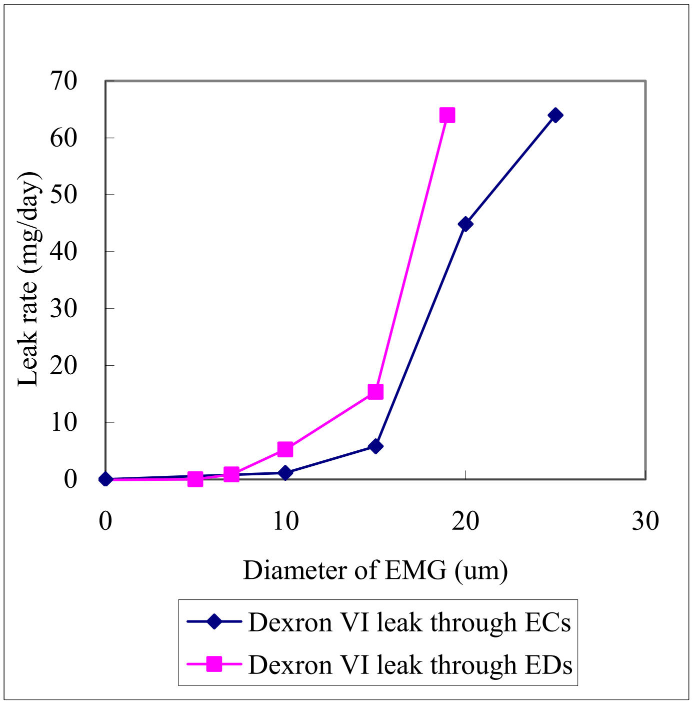

Figure 4: Total leak rate of Dexron® VI through ECs and EDs of different diameters. Leak rate is strongly dependent on diameter of the EMG.

A typical small drop of Dexron® VI, depending on the drop-formation mechanism, weighs 5 – 20 mg. Assuming that the maximum allowed total leak is 5mg/day, the maximum allowed EMG would be a 7-μm ED or a 15-μm EC (with a nominal length of 3.5 mm). The 10-μm ED showed greater variability and hence, a 7-μm ED is a conservative choice.

The nitrogen flow rate through a 7-μm ED is approximately 0.4 cc/min at 15-psig and flow through a 15-μm EC is approximately 0.09 cc/min at 15-psig. Since the airflow associated with the 15-μm EC (3.5 mm nominal length) is less than a 7-μm ED, a 15-μm EC of 3.5 mm length would be a tight leak specification.

This exercise also proves that oil leak rate does not bear a direct correlation with airflow rate. Though the airflow through a 15-μm EC was significantly less than a 10-μm ED, the oil leak rate is almost identical.

During a production leak test, gas (air or helium) flow is used to set up the acceptance criteria. As a 7-µm ED and 15-µm EC are the “acceptable” micro-geometries, it is clear that the maximum allowed gas leak, during production leak testing, should be set based on the EC flow. If the maximum allowed flow is set on the 7-µm ED at 15 psig (0.4 cc/min), ECs with diameters of 25-µm and above will pass, resulting in oil leaks in the field. Therefore, selecting leak tightness specification based on EC covers potential leaks from EDs. Selecting ED as a leak tightness specification may cause parts with larger ECs to pass.

Nitrogen flow test through the EMGs applied at the same direction as oil pressure, at 15-psig, 80-psig and 150-psig, at the end of the test revealed that only 3 EMGs of the 26 used were not completely clogged. The 3 EMGs were a 22-µm ED, a 20-µm EC and a 25-µm EC. Figures 5 and 6 present a comparison of nitrogen flow rates at 15 psig, through Equivalent Channels and Equivalent Diameters, before and after the test.

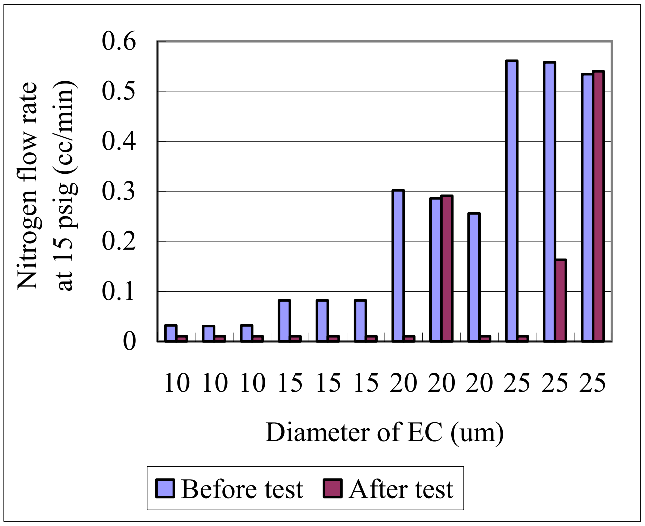

Figure 5: Comparison of nitrogen flow rates through Equivalent channels before and after they were exposed to Dexron® VI at 300-psig and 300 F

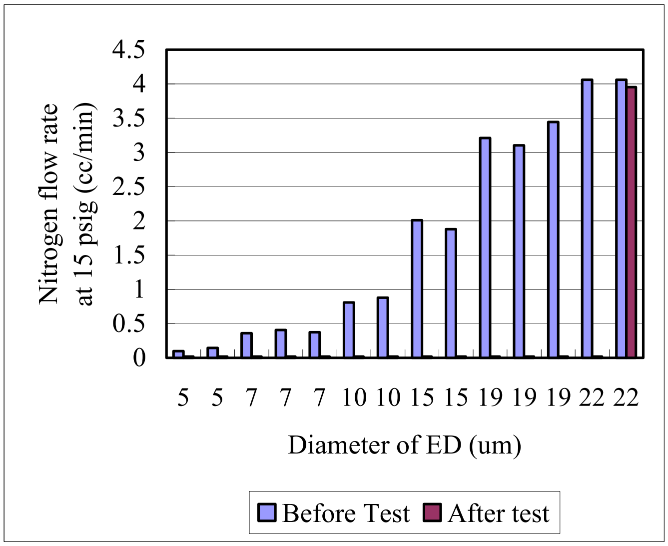

Figure 6: Comparison of nitrogen flow rates through Equivalent diameters before and after they were exposed to Dexron® VI at 300-psig and 300 F

It was also observed, especially in EDs, that most leak occurred in the first 24 hours of the test, after which the EMGs got clogged.

To verify if the EMGs would open up again, an oil pressure ‘shock’ of 300-psig was applied. But it failed to open the orifices. This led us to believe that once the EMGs are clogged, they tend to stay clogged. This test was conducted in a controlled lab environment. In real life applications, there will be frequent temperature and pressure spikes that may or may not have an affect on EMG clogging. Therefore, a conservative approach in selecting the acceptable EMG, for production leak tightness specification, has been used.

APPLICATION OF LEAK TIGHTNESS SPECIFICATION FOR PRODUCTION LEAK TESTING

The selected EMG configuration for production leak tightness specification has the additional advantage of being independent of the leak testing technology. The selected method for leak testing, the gas used and the test pressure are entirely functions of the available test times and cost. Care must be taken, though, that the test pressure does not “mask” leaks due to component limitation.

As suggested, selecting an EC provides a safer approach and hence, in this case an EC of 15 micron and 3.5 mm long was chosen as the leak tightness specification for protection against oil leaks in the field.

An example is presented here that illustrates the use of a microflow technology, using air as the test fluid (9,10,11). The unique gas microflow sensor, called the Intelligent Gas Flow Sensor or IGLS, is capable of measuring gas flow rates in flow regimes ranging from viscous flow to molecular flow. The test method is based the law of conservation of mass. The IGLS measures the amount of flow that is required to keep a unit under steady pressure. A detailed description of the patented micro-flow sensor is available in the literature (9). The sensor is set to measure volumetric leak rate (in cc/min) and plot a flow rate versus time (or signature) during the test. A typical leak test cycle has three steps. The senor is placed between a pressure source and the part being tested. First, during the Fill stage, the part being tested is pressurized to the test pressure, while the sensor is shunted. Second, during the Stability stage, the sensor is introduced in the circuit and flow from the pressure source to the leaking part occurs through the sensor. The sensor measures the amount of flow required to maintain the part at a steady pressure. At steady state condition, the leak rate vs. time is nearly a flat line. Last, during the test stage, the sensor compares the flow rate to the accepted flow rate and makes a decision on whether to accept or reject the part.

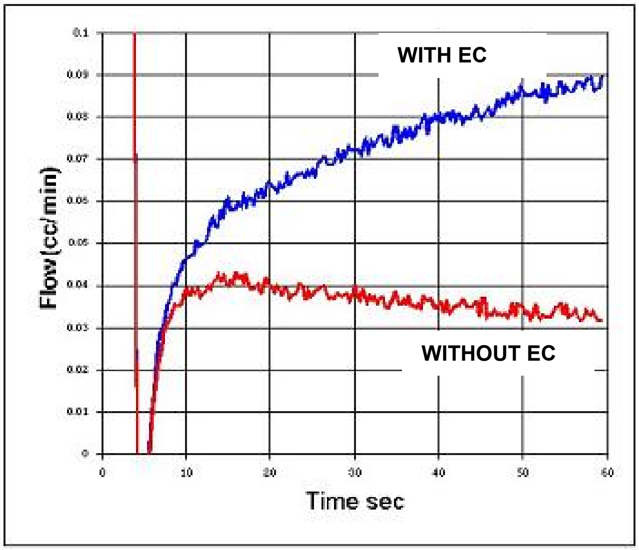

Figure 7: Comparison of test signatures of a transmission component with and without a leak of 15-μm by 3.5 mm EC (Measured by Sensor P/N IL-001C-100S with a measurement range of 0-1 cc/min).

Figure 7 presents test signatures of an automotive transmission component, a machined aluminum casting, subjected to leak tightness specifications of 15-μm diameter, 3.5 mm long EC. The bottom signature belongs to a non-leaking master part while the top signature belongs to the same part, plus a 15-μm EC attached to it as a leak simulator. The acceptance criterion, for parts in production, is set based on these dynamic signatures.

As can be seen from figure 7, the final reading of the leaking part is almost 3 times that of a non-leaking part. The total test time is approximately 60 seconds. The ‘master’ part exhibits a leak rate of 0.03 cc/min at 15 psig, while the same part with an attached EC measures 0.09 cc/min @ 15 psig. Therefore, for every subsequent production test part, the maximum allowed leak rate for this specific set up should be under 0.09 cc/min. The test time and maximum accepted air leak can be adjusted for a shorter cycle time, as long as there is an acceptable difference between a non-leaking part and a leaking part. A 2:1 ratio between an accepted non-leaking part and the same part with an EC is a guarantee that the part leak tightness, during that leak test time, is better than the maximum allowed EC. If the part maintains the same seal integrity as it had displayed during the leak test, it is highly likely that there would no external oil leak.

Once the initial setup is established, a group of parts is tested multiple times to establish the measurement repeatability and capability.

CONCLUSION

The complexity of transferring operational leak tightness requirements of various liquid and vapor applications into a realistic production leak test specifications can be overcome by using an empirical concept, described as the Equivalent Micro Geometry concept. The empirical tests are quite simple, as described in the case of automatic transmission fluid, and a generic conclusion of maximum allowed Equivalent Channel or Equivalent Diameter geometry can be drawn. During a product’s functional life, it does not sustain continuous leak (fluid or vapor) through such geometries, due to fluid non-homogeneity and contaminations, surface tension, capillary forces and micro-electrostatic boundary layers. Meeting the leak tightness of such an EMG will assure no emission or contamination due to leakage. Hence, the existence of multiple EMGs in a product is not of concern since they have a tendency to get clogged after exposure to fluid for a short time. The data suggests that selecting an Equivalent Channel (EC) as the leak tightness specification, rather than a sharp edged geometry (ED), results in a tighter air leak specification and increases the possibility of catching all types of EMGs during production leak testing.

Once the Equivalent Channel range is empirically defined, it becomes the production leak tightness specification. The exact leak mechanism through the EMGs requires further investigation. Slip effects, surface tension effects; oil additives and contaminants, capillary forces and micro-electrostatic boundary layers are some of the prominent mechanisms that affect leak rate.

The leak test method may be an air microflow technology, as described, or any other capable leak tightness method, as long it can distinguish between a good master part and the same part with an EC. The leak tester is actually a comparator of any production part to a master part and a master part with an EC attached. The total leak test time can be optimized to allow dynamic signature testing as long as, at the end of the test, a significant ratio between set of master parts and the same parts with EC attached exists. A minimum factor of 2:1 is recommended, but can be decided based on the application.

ACKNOWLEDGMENTS

This work was performed at the Advanced Test Concepts (ATC) Test and Metrology Laboratory with the support of Mr. Rana Ghosh and Mr. Paul Traub. The support and feedback of Mr. Edmund Sieracki of GM Powertrain Engineering and Mr. Bruce Essex of GM Powertrain Allison was crucial to complete this research work. The test results and work published by all participants, is an engineering contribution to enhance efforts toward global environmental and safety standards.

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