Development of an Ignition Method for the Self-decomposition of HFO Refrigerants

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ABSTRACT

Hydrofluoroolefin (HFO) refrigerants have emerged as next-generation refrigerants; however, their tendency to undergo self-decomposition reactions raises safety concerns. For the widespread commercialization of such refrigerants, it is essential to clarify the conditions under which they can be used safely. However, standardized evaluation methods have not yet been established. This study aims to address this gap by focusing on ignition method—the most critical component of the evaluation process—and developing a discharge method that enables energy quantification while simulating discharge phenomena occurring in actual equipment that triggers self-decomposition. Furthermore, we investigate how different ignition methods influence the propagation of self-decomposition reactions and evaluate the relationship between the fusing wire and arc discharge method and the newly developed discharge method.

Keywords: HFO Refrigerants, Self-decomposition, Ignition method, Discharge method

BACKGROUND

In recent years, environmental regulations on refrigerants in the refrigeration and air conditioning sector have tightened due to increasing global concern over greenhouse gas emissions. Under the Kigali Amendment to the Montreal Protocol, a global consensus has emerged to phase down high-global warming potential (GWP) hydrofluorocarbons (HFCs) and transition to environmentally friendly alternatives such as natural refrigerants and hydrofluoroolefins (HFOs).

Although these next-generation refrigerants exhibit significantly lower GWP than conventional ones, they present safety challenges, including flammability, toxicity, and chemical instability. In particular, HFOs are less chemically stable due to the presence of carbon–carbon double bonds in their molecular structure. Reports indicate that under extreme conditions, such as high temperature and pressure, these compounds can undergo energy-triggered self-decomposition, potentially leading to the propagation of chain reactions [1–3]. These safety risks hinder their widespread commercialization.

To enable the safe use of such refrigerants, it is essential to define the operational conditions under which they can be used without risk. However, standardized evaluation methods for these conditions have not yet been established.

This study focuses on ignition methods, a critical component in risk evaluation. We developed a discharge-based ignition method that quantifies the discharge energy required to initiate self-decomposition while simulating discharge phenomena observed in actual refrigerant compressors. Furthermore, we examined the influence of different ignition methods on the propagation of self-decomposition and clarified the correlation between the fusing wire and arc discharge method and the newly developed approach.

SELF-DECOMPOSITION REACTIONS

A self-decomposition reaction is a chemical process in which a single compound breaks down into multiple distinct products. This process often involves radical chain propagation and typically results in the formation of structurally rearranged species.

Driven by heat or energy input, the unidirectional fragmentation of molecules frequently produces smaller fragments, radicals, and gaseous byproducts. Accordingly, such reactions are also known as thermal decomposition reactions.

Self-decomposition is not uncommon. In particular, unsaturated fluorinated refrigerants containing carbon–carbon double bonds (C=C) or weak carbon–halogen bonds are prone to self-decomposition under abnormal conditions—such as elevated temperature and pressure—when triggered by stimuli like electrical discharge. These events can initiate chain reactions that propagate decomposition.

Understanding the thermal behavior of such refrigerants is essential for ensuring the safety and reliability of air conditioning systems.

Representative self-decomposition reactions of known compounds are presented below:

- Tetrafluoroethylene (TFE, $CF_2=CF_2$):

$$CF_2=CF_2 \rightarrow CF_4 + C \qquad \Delta H_R = -257\ \text{kJ/mol}$$

- Trifluoroethylene (HFO-1123, CF_2=CHF):
  CF_2=CHF → 0.5 CF_4 + 1.5 C+HF $\Delta H_R = -250$ kJ/mol
- Trans-1,2-difluoroethylene (R-1132(E), trans-CHF=CHF):
  CHF=CHF → 2C(s) + 2HF $\Delta H_R = -248$ kJ/mol

Given these characteristics, it is essential to develop evaluation methods to determine the conditions under which self-decomposition reactions of refrigerants propagate to the surrounding environment—both for next-generation refrigerant development and for system design.

Even R32, a saturated compound, can undergo self-decomposition when triggered by arc discharge or metal wire fusing, producing carbon and hydrogen fluoride (HF). However, due to its higher thermal stability and lower decomposition reactivity compared to unsaturated compounds, the reaction generally does not propagate.

Therefore, in assessing chemical stability, the focus should not be solely on whether self-decomposition occurs upon ignition, but rather on whether the reaction propagates under the specified test conditions.

Evaluation Method for Self-Decomposition Reactions and Development of Ignition method

The chemical stability of refrigerants has previously been evaluated using stainless steel or Inconel pressure vessels equipped with internal ignition sources, as reported in previous studies [1–4]. In this study, we evaluated the threshold conditions beyond which self-decomposition reactions begin to propagate, using the test apparatus shown in Fig. 1.

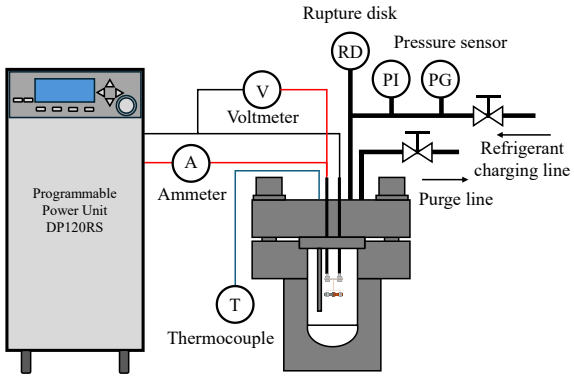

Fig. 1 Schematic of experimental apparatus.

Among various evaluation methods, ignition methods are the most critical. In particular, it is necessary to develop ignition techniques that simulate *layer-short* phenomena, which are considered the actual triggers of self-decomposition reactions in operating equipment. Ignition methods can be broadly categorized into fusing wire and discharge types.

The thin metal wire fusing method is widely used due to its simplicity; however, it fundamentally differs from arc discharge phenomena—which involve high-temperature plasma and are more representative of real-world triggers. Therefore, there is a growing demand for discharge-based ignition methods.

Standardized refrigerant flammability evaluation methods, such as ASTM E681, and the study by Goto et al. (2024), generate arc discharges through electrical breakdown by applying extremely high voltages between electrodes. However, according to Paschen's Law, initiating such discharges under the high-pressure conditions found in actual refrigerant compressors remains a significant challenge [4],[5].

Compared to breakdown-based discharges, fusing methods are more stable under high-pressure atmospheres. Previous studies have proposed ignition methods capable of generating arcs under high pressure by applying AC or DC voltages to electrode structures with physical contact points, enabling arc formation via microscale gap separation [2],[3]. However, these methods require extensive trial-and-error and present difficulties in quantifying energy, particularly for AC discharges.
Therefore, developing ignition methods that combine simplicity with realistic simulation of discharge phenomena in actual equipment has become an urgent priority. Additionally, ignition techniques capable of supporting multiple discharges are desirable to improve experimental efficiency.

The AC discharge method using tungsten electrodes poses challenges in energy quantification, while the DC discharge method with copper electrodes demands considerable operator experience and still faces issues with multiple consecutive ignitions.

To address these challenges, we developed a DC discharge method based on a contact–separation electrode structure, as shown in Fig. 2, featuring a novel electrode material design. The electrodes combine a high-melting-point tungsten bolt, which ensures thermal stability, with a low-melting-point copper bolt, which facilitates the generation of metal vapor necessary for plasma formation. This design enables both reliable discharge initiation and resistance to electrode sticking.

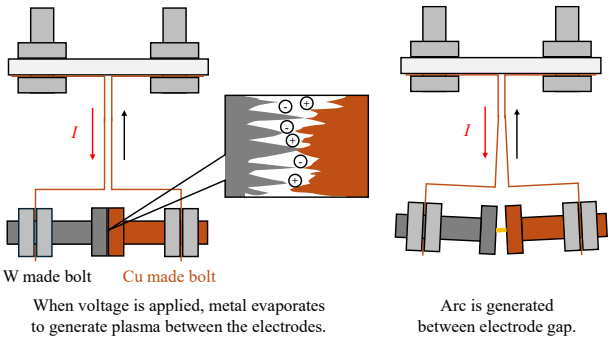

Fig.2 Mechanism of contact-separation electrodes.

The discharge characteristics of the DC discharge

method in air are shown in Fig. 3. While arc voltage is known to depend on parameters such as the first ionization energy of the electrode material, electrode gap, and pressure, the use of a contact–separation electrode structure implies that arc formation primarily depends on the electrode material, as the gap at the moment of separation is extremely small.

Both tungsten and copper have first ionization energies of approximately 7.8 eV. The measured arc voltage (after subtracting wiring resistance) remained stable at around 15 V, as experimentally confirmed. Since the resistance between electrodes is negligible during contact, applying voltage causes a short circuit, resulting in current reaching the limiter value of the power supply. By adjusting the current limiter, the energy density can be controlled.

Discharge energy measurements under a 3 MPa R32 atmosphere, with current limiter values set to 150 A and 378 A, are shown in Fig. 4 and Fig. 5. These results demonstrate that even under high-pressure gas conditions, discharge energy can be quantitatively evaluated as a function of voltage application duration across different energy density settings.

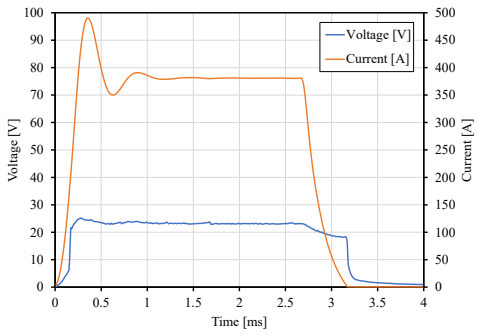

Fig. 3 I/V waveforms vs. time (378 A limiter).

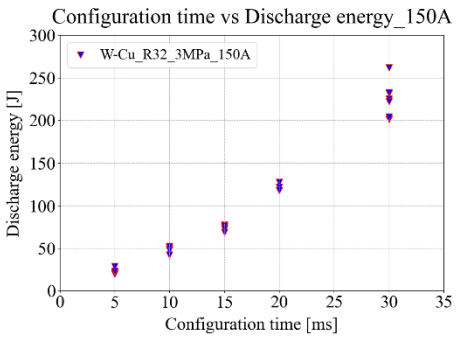

Fig. 4 Discharge energy quantification at 3.0 MPa R32 (150 A limiter).

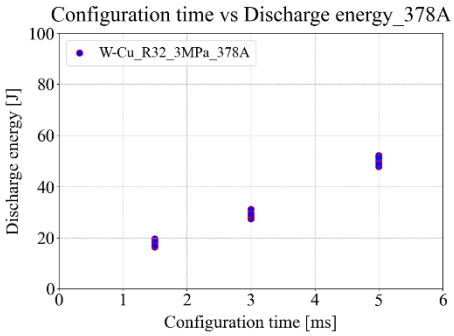

Fig. 5 Discharge energy quantification at 3.0 MPa R32 (378 A limiter).

BOUNDARY CONDITIONS OF HFO-1123-BASED TERNARY REFRIGERANT MIXTURES

Using the DC discharge method described above, we evaluated both the minimum energy threshold required for the propagation of self-decomposition reactions and the maximum allowable HFO-1123 mixing ratio in ternary refrigerant mixtures composed of HFO-1123, R32, and R1234yf (mass ratio: x/21.5/78.5 – x). The tests were conducted under assumed typical operating conditions for air conditioning systems: 130 °C and 4.0 MPa. The results are presented in Fig. 6.

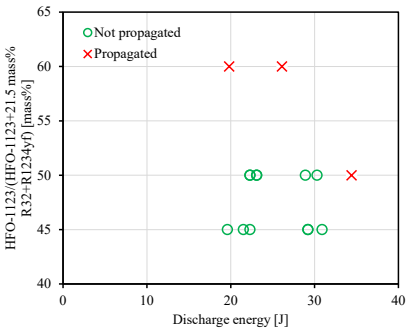

Fig. 6 Experimental results varying HFO-1123 concentration and discharge energy.

The results indicate that even at HFO-1123 concentrations up to 50 mass%, the minimum energy required to propagate self-decomposition remained above 30 J. However, at 60 mass%, the energy threshold dropped below 20 J, suggesting an increased risk of reaction propagation at higher HFO-1123 concentrations in the ternary mixture.

COMPARISON OF IGNITION METHODS

As part of the ongoing NEDO project (P23001), a hybrid ignition method combining the simplicity of wire fusing with the high reproducibility of arc discharges has been proposed. This method simulates layer-short phenomena, which are believed to trigger self-decomposition reactions in actual equipment.

Within the scope of this project, we investigated the influence of various ignition methods on the boundary pressure for the propagation of self-decomposition reactions. 1,1-Difluoroethylene (R1132a, also known as VDF) was selected as a reference substance, and four ignition methods were evaluated:

- AC discharge (contact–separation electrodes)
- DC discharge (contact–separation electrodes)
- Ni fusing wire and discharge
- Cu fusing wire and discharge

Tests were conducted at 150 °C (100 °C for AC discharge), with discharge energies in the range of 20–30 J. The resulting boundary pressures for self-decomposition propagation are shown in Figs. 7–10. According to Lisochkin and Poznyak (2006), the boundary pressure for R1132a self-decomposition propagation at 100 °C is approximately 3.0 MPa [1].

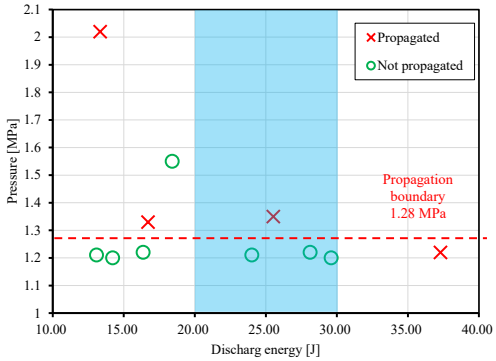

Fig. 7 Experimental results using the Ni fusing wire and discharge method.

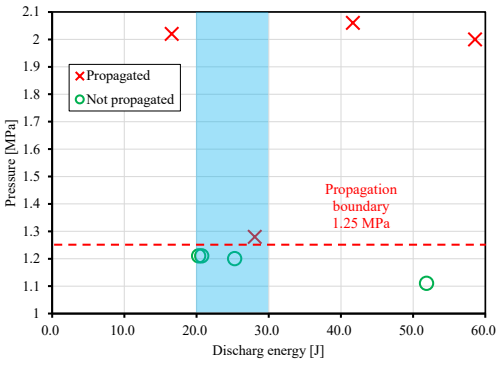

Fig. 8 Experimental results using the Cu fusing wire and discharge method.

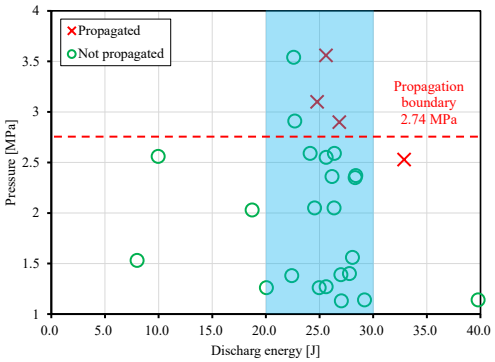

Fig. 9 Experimental results using the DC discharge method.

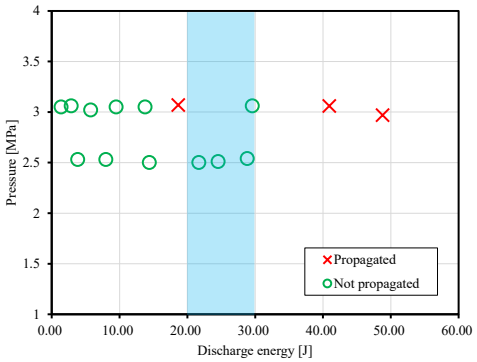

Fig. 10 Experimental results using the AC discharge method (at 100 °C).

The results of the fusing wire and arc discharge method tests revealed boundary pressures of 1.28 MPa and 1.25 MPa for Ni and Cu wires, respectively, indicating that the wire material had minimal influence. In contrast, the AC and DC discharge methods using contact–separation electrodes exhibited significantly higher boundary pressures, ranging from 2.5 to 3.0 MPa. These findings suggest that electrode structure plays a dominant role in determining the boundary pressure, likely due to differences in thermal energy transfer.

In the fusing wire and arc discharge method, the electrode gap is approximately 1 mm, enabling efficient energy transfer from the arc to the surrounding gas. Conversely, in the contact–separation structure, arcs are formed in narrow gaps created after contact separation, but the bolt-shaped electrode tips result in thermal losses through heat conduction, thereby limiting energy transfer to the gas. Consequently, self-decomposition reactions are considered to propagate only when the transferred energy exceeds a threshold value—similar to the minimum ignition energy required for combustion. Otherwise, only localized initiation occurs without propagation.

Since layer-short phenomena—the presumed trigger for self-decomposition—typically occur between

windings or at terminal connections inside refrigerant compressors, heat loss at these locations cannot be neglected. Therefore, to enable realistic and non-conservative evaluation of refrigerant stability, electrode design must account for thermal losses at sites where layer-short events are likely to occur.

CONCLUSION

In this study, we developed a DC discharge ignition method incorporating a novel electrode material design that fulfills the following criteria:

- high reproducibility of layer-short phenomena (considered actual decomposition triggers),
- capability for quantitative measurement of ignition energy, and
- potential for multiple consecutive ignitions to improve test throughput.

To validate this evaluation approach, we examined the influence of different ignition methods on self-decomposition propagation. The results demonstrated that electrode structure significantly affects the boundary pressure, highlighting the importance of thermal loss associated with structural variation.

These findings emphasize the necessity of considering the thermal environment at locations prone to layer-short events when designing electrode structures for evaluating the chemical stability of refrigerants.

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HFO 冷媒の自己分解反応を評価するための着火法の検討

Development of an Ignition Method for the Self-decomposition of HFO Refrigerants

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Hydrofluoroolefin (HFO) refrigerants have emerged as next-generation refrigerants; however, their tendency to undergo self-decomposition reactions raises safety concerns. For the widespread commercialization of such refrigerants, it is essential to clarify the conditions under which they can be used safely. However, standardized evaluation methods have not yet been established. This study aims to address this gap by focusing on ignition method—the most critical component of the evaluation process—and developing a discharge method that enables energy quantification while simulating discharge phenomena occurring in actual equipment that triggers self-decomposition. Furthermore, we investigate how different ignition methods influence the propagation of self-decomposition reactions and evaluate the relationship between the fusing wire and arc discharge method and the newly developed discharge method.

Key Word: HFO Refrigerants, Self-decomposition, Ignition method, Discharge method

１．背景

近年冷凍空調の分野では、温室効果ガス削減への関心の高まりから、冷媒に対する環境規制が強化されている。国連では温室効果が懸念されるハイドロフルオロカーボン（HFC）冷媒から自然冷媒やハイドロフルオロオレフィン（HFO）冷媒のような低 GWP 冷媒への転換が要求されている。これらの新しい冷媒は従来冷媒に比べて温室効果が大幅に削減されているが、可燃性、毒性、化学的な不安定性のような安全面の課題がある。HFO 冷媒は分子構造内に二重結合を持つため化学的に不安定である。高温高圧のような過酷な条件下でエネルギーが与えられると自己分解反応が伝播することが報告されており [1), 2), 3)]、実用面で安全性の懸念がある。このような冷媒を普及するには安全に使用できる条件範囲を明確にする必要があるが、その評価手法はまだ規格化されていない。

本研究では、評価手法の中でも最も重要とされる着火法に着目し、自己分解反応の発生誘因である実機での放電現象を模擬した、エネルギーの定量化が可能な放電法を開発した。異なる着火法が自己分解反応の伝播に及ぼす影響についても調査し、金属細線の溶断放電法と放電法の相関関係を明らかにした。

２．自己分解反応

自己分解反応とは、単一の化合物が分解して複数の異なる生成物を生じる反応を指す。このプロセスはラジカルの連鎖伝播によって進むことがあり、構造が再編成された化学種を生成する場合が多い。温度上昇やエネルギー入力により分子が一方向に分解し、より小さな断片やラジカル、気体副生成物を生じるという共通点があることから熱分解反応ともよく呼ばれる。

自己分解反応は決して珍しい現象では無い。特に炭素–炭素二重結合（C=C）や弱い炭素–ハロゲン結合を含む不飽和フッ素化冷媒は、異常運転時に至る高温高圧の状態、放電のような発生誘因があると自己分解反応を誘発し、周囲への伝播に繋がる可能性がある。これら冷媒の熱挙動を理解することは、空調システムの安全性と適合性を確保する上で不可欠である。

以下の物質は自己分解反応を起こすことが知られており、それぞれの反応式を以下に示す：

- Tetrafluoroethylene (TFE, $CF_2=CF_2$):

$CF_2=CF_2 \rightarrow CF_4 + C$ $\Delta H_R = -257$ kJ/mol

- Trifluoroethylene (HFO-1123, $CF_2=CHF$):

$CF_2=CHF \rightarrow 0.5\ CF_4 + 1.5\ C + HF$ $\Delta H_R = -250$ kJ/mol

- Trans-1,2-difluoroethylene (R-1132(E), trans-CHF=CHF):

$CHF=CHF \rightarrow 2C(s) + 2HF$ $\Delta H_R = -248$ kJ/mol

このような特性から、冷媒の開発やシステム設計において自己分解反応が周囲に伝播する条件範囲の評価手法を開発する必要がある。飽和化合物である R32 は、アーク放電や細い金属線の溶断を誘因として炭素やフッ化水素（HF）を生成する熱分解を起こすことがあるが、不飽和化合物とは異なり、熱安定性が高く分解エネルギーが小さいため、分解が周囲に伝播することは通常ない。したがって、化学的な安定性の評価では着火により自己分解反応の発生ではなく、試験条件下で周囲へ伝播するかどうかに重点を置くべきである。

３．自己分解反応の評価手法及び着火法の開発

冷媒の化学的な安定性は着火源を内蔵したステンレス製やインコネル製の圧力容器を用いて実施されていることが報告されている [1-4]。本研究では Fig. 1 に示しているような試験装置を用いてして自己分解反応が伝播する境界条件を評価した。

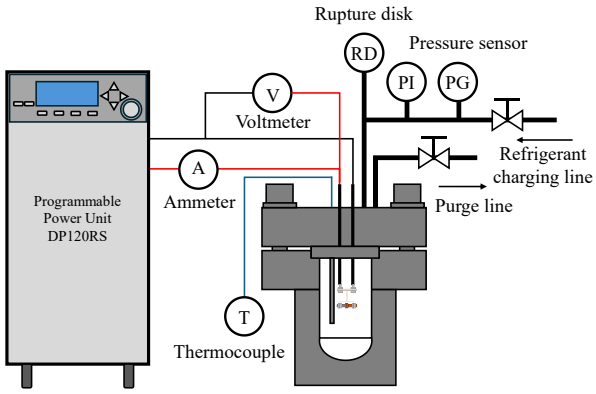

Fig. 1 Schematic of experimental apparatus.

評価手法の中で最も重要なのは着火法であり、実機での自己分解反応の発生誘因であるレイヤーショート現象を模擬した着火法の開発が求められる。着火法は大きく溶断法と放電法に分けられる。一般的には簡便性が高い金属細線の溶断法で実施されているが、金属が融解して周囲のガスにエネルギーを伝える現象と実機での発生誘因であるアーク放電のような高温のプラズマを生成する放電現象とは異なるため、放電法の開発が求められる。

冷媒の燃焼性を評価する国際規格 ASTM E681 と Goto et al. (2024)では高電圧の印加による絶縁破壊により電極間で放電させる方法を使用しているが、Paschen's law により自己分解反応が起こりうる冷媒圧縮機内の高圧雰囲気中で起こすことが課題になる [4), 5)]。絶縁破壊による放電法と比べて金属細線の溶断法は高圧雰囲気中でも安定的に起こすことができる。先行研究では接点を持つ電極構造に AC・DC 電圧をかけることで電極が解離して、微小の電極間の隙間にアークを発生させる方法で高圧雰囲気中でも放電を起こす着火法が紹介されているが、実現するには事前の試行錯誤が必要な点やエネルギーの定量化（交流放電法）の課題がある [2), 3)]。そのため簡便性と実機現象の再現性を両立した着火法が求められている。上記以外に試験の生産性を上げる連続放電を可能とした着火法の開発が望ましい。

タングステン電極を使用した交流放電法はエネルギーの定量化の課題があり、銅製電極を使用した直流放電法では経験と試行錯誤が必要になる点と連続放電の課題がある。そのため本研究では電極の材質を工夫した Fig. 2 に示した構造の接点解離式電極による直流放電法を開発した。電極の先端部は高融点のタングステンとプラズマを

生成するのに不可欠な金属蒸気を生成しやすい低融点の銅製のボルトを使用することで放電の起き易さと比較的な難溶着性を両立した着火法を開発することができた。

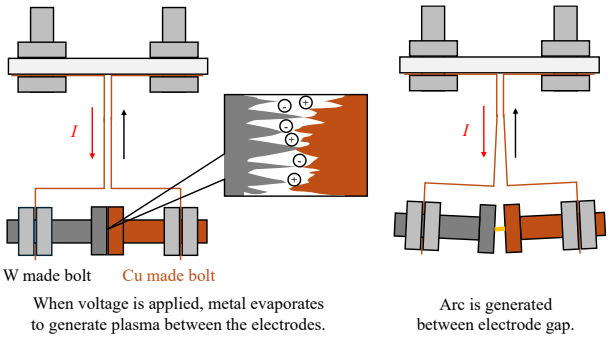

Fig.2 Mechanism of contact-separation electrodes.

大気中の直流放電法で試験した時の放電特性を Fig. 3 に示す。アーク電圧は電極材質の一次電離電圧と電極間距離及び圧力に依存することが報告されているが、接点を持つ電極のため、電極がプラズマによりアークが生成され、電極離れた時の隙間は極めて小さく、ほぼ電極の材質のみに依存することになる。タングステンと銅の一時電離電圧は約 7.8 Vのため、配線抵抗を差し引いたアーク電圧が約 15 V一定になっていることを実験で検証することができた。電極間の抵抗が極めて小さいため、電圧を掛けた時はショート現象になるため、電流は電源装置のリミッター値になり、電流リミッターを制御することでエネルギー密度を制御することができる。3 MPa の R32 雰囲気中での電流リミッターが 150 Aと 378 Aの条件で放電エネルギーの定量化を評価した結果を Fig. 4 and 5 に示す。結果からは 3 MPa のガス雰囲気中でも二種類のエネルギー密度の条件で放電エネルギーを電圧の印加時間で定量化することができた。

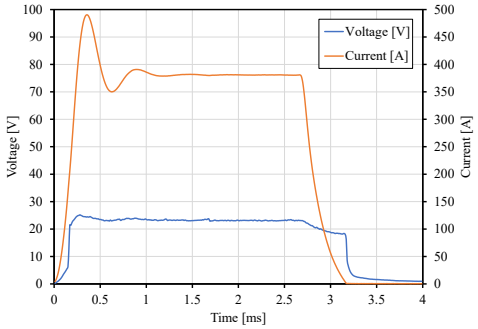

Fig. 3 I/V waveforms vs. time (378 A limiter).

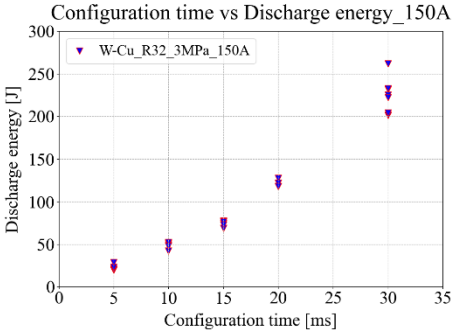

Fig. 4 Discharge energy quantification at 3.0 MPa R32 (150 A limiter).

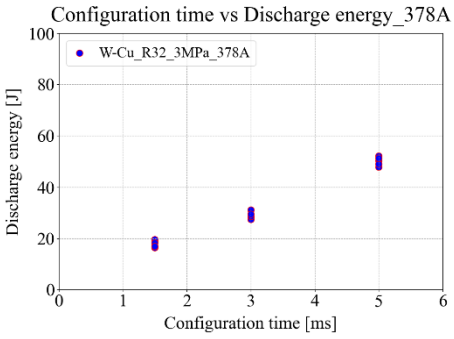

Fig. 5 Discharge energy quantification at 3.0 MPa R32 (378 A limiter).

4. HFO-1123 混合冷媒の伝播境界条件の評価

上記で紹介した直流放電法で HFO-1123 を含んだ三元系混合冷媒（HFO-1123/R32/R1234yf＝*x*/21.5/78.5-*x* mass%）の自己分解反応が伝播する境界エネルギー量と HFO-1123 の最大混合比を評価した。試験条件はエアコンの通常運転条件を

想定し、130 ℃と 4.0 MPa に設定した。試験結果を Fig. 6 に示す。試験結果から HFO-1123 を 50 mass%まで混合しても、自己分解反応が伝播するエネルギー境界は 30 J 以上であることが分かった。混合率が 60 mass%の場合はエネルギー境界が 20 J 以下出ることが分かった。

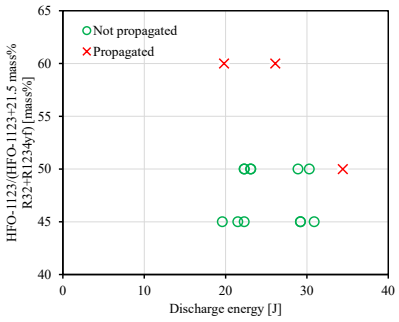

Fig. 6 Experimental results varying HFO-1123 concentration and discharge energy.

５．異なる着火法の比較

今期の NEDO プロジェクト（P23001）では溶断法の簡便性と放電法の実機での発生誘因であるレイヤーショートの高い再現性を両立した金属細線の溶断放電法が提案されており、本研究ではその一環として、異なる着火法が自己分解反応の伝播境界条件に与える影響について調査した。

標準物質として R1132a（ＶＤＦ）を選び、接点解離式電極を使用した AC・DC 放電法と、異なる金属細線（Ni と Cu）を使用した金属細線の溶断放電法の四種類の着火法で、温度条件が 150 ℃（交流放電法は 100 ℃）と着火エネルギーが 20-30 J の時の自己分解反応の伝播境界圧力について評価した結果を Fig. 7-10 に示す。Lisochkin, Y. A., and Poznyak, V. I. (2006) は 100℃の時の R1132a の自己分解反応の伝播境界圧力は 3.0 MPa 付近にあることが報告されている [1)]。

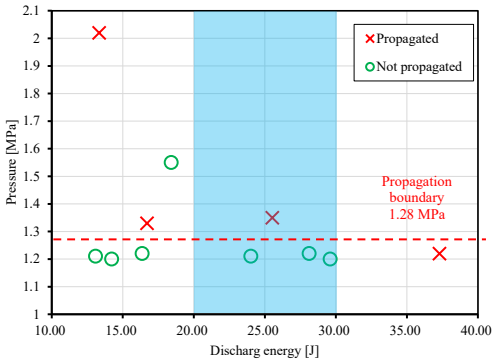

Fig. 7 Experimental results using the Ni fusing wire and discharge method.

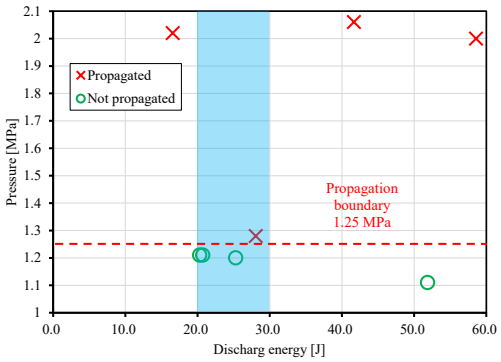

Fig. 8 Experimental results using the Cu fusing wire and discharge method.

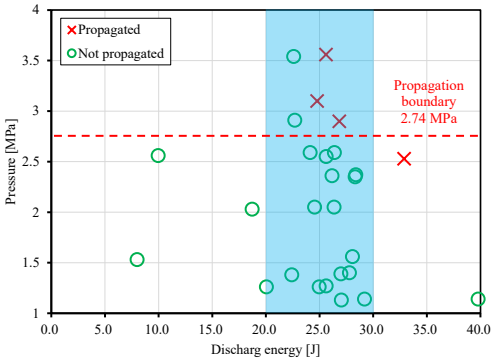

Fig. 9 Experimental results using the DC discharge method.

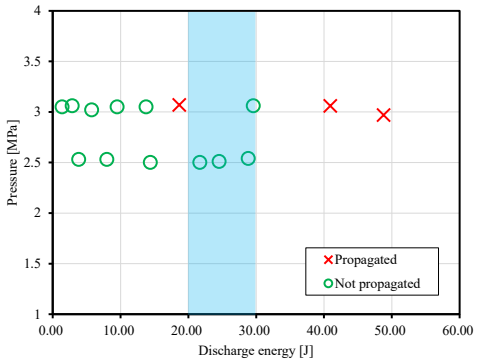

Fig. 10 Experimental results using the AC discharge method (at 100 ℃).

溶断放電法の結果から、金属細線の材質をNiとCu で評価し他結果、自己分解反応が伝播する境界圧力はそれぞれ 1.28 と 1.25 MPa であり、Ni と Cu では材質の影響は小さいことが分かった。接点解離式電極を使用した AC・DC 放電法で試験した結果、自己分解反応が伝播する境界圧力は 2.5 から 3.0 MPa の間にあることが分かった。上記の結果から、溶断放電法と接点解離式電極で境界圧力が大きく異なり、原因は電極構造にあることが分かった。溶断法放電法は電極間距離が 1 mmであり、アーク生成によって投入されたエネルギーは十分周囲のガスに伝わることができる。それに対して、接点解離式電極では先端部がボルトでできており、電極が離れて極めて小さい隙間にアークを生成しても、先端部の伝熱によりロスで一部のエネルギーが周囲のガスに伝わらないことが原因になることが考えられる。ガスに伝わったエネルギーが自己分解反応が伝播するのに必要な最小伝播エネルギーを超えると反応が伝播し、それを満たさない場合は初期反応が起きるが、反応の伝播が抑制されることが考えられる。自己分解反応が起きる発生誘因であるレイヤーショートは冷媒圧縮機内の巻き線間や端子ターミナルで起きるため、同様に伝熱の影響は無視できない。そのため、冷媒の化学的な安定性を過酷に評価しないには、実機で起きる現象を再現する電極構造を設計する際にレイヤーショートが発生しうる箇所の伝熱による熱ロスの影響を考慮する必要があることが分かった。

６．結論

本研究では冷媒の化学的な不安定性による自己分解反応の評価手法を規格するために、発生誘因であるレイヤーショート現象の再現性、着火エネルギーの定量化と生産性を上げるための連続放電を同時に満たす直流放電法を開発した。評価手法の妥当性について、異なる着火法の影響について調査し、電極構造が異なると境界圧力が大きく異なることが分かった。この結果は電極構造の相違で起きる伝熱ロスの影響を示唆しており、実機でレイヤーショートが起きる箇所の構造も電極構造を設計する際に考慮する必要があることが分かった。

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謝辞

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