Neviere 等[30]采用动态机械分析仪(DMA)研究了HTPB固体推进剂的老化行为。第二个峰的tgδ反映了粘合剂的氧化,并与拉伸强度成线性相关关系,显示了DMA是测量由固体推进剂老化所引起损伤的强大工具。Husband[31]用动态粘弹法研究了火箭发动机中的样品和方坯样品的动态贮存模量 G' 与贮存温度和时间的关系,进而得出固体推进剂老化速率与温度的关系。Duncan等[32-33]用单轴拉伸法和动态粘弹法,研究了AP/HTPB固体推进剂的单向抗张模量 E_r 和动态贮能模量 G' 的相关性。试验结果表明,当动态剪切应变为2.0%时,在实验温度和应变速率范围内,G' 和 E_r 有很好的相关性。

Gottlieb[34]研究了HTPB推进剂中增塑剂己二酸二辛酯(DOA)的迁移对推进剂性能的影响。他认为增塑剂的迁移可分为两个阶段:固化过程中的迁移和贮存老化过程中的迁移。在固化过程中,三维网状结构形成之前,可能存在增塑剂的迁移速率较大。在老化过程中,DOA向衬层/绝热层的迁移的扩散系数较小,比同样条件下DOA从推进剂本体向外迁移的扩散系数小15倍。在贮存老化过程中,增速剂迁移达到平衡前,对药柱的力学性能有明显影响,同时还会引起发动机粘接界面性能的变化,从而破坏发动机药柱的结构完整性。Judge等[35]采用近红外光谱(NIR)测试了推进剂组分中的防老剂和增塑剂。NIR是具有较高可靠性和精密度的方法,对初始配方的测试含量与实际含量非常吻合,该测试方法可用于贮存过程中防老剂和增塑剂的监测。

粘合剂基体和氧化剂等固体填料的"界面"老化即"脱湿",是推进剂应力应变发生变化的重要原因。"脱湿"现象是一个过程,随着"脱湿"现象的发生,分散相和连续相之间的物理吸附或化学吸附力降低,或附加交联破坏,使整个体系内的应力传递遭到削弱,于是填料的补强效果很快下降。Rothon[36]的研究表明,对于颗粒增强复合材料其界面"脱湿"是导致材料损伤破坏的主要形式之一。Bellerby等[37]采用键和剂来预防硝胺和粘合剂的界面"脱湿"。Hubner等[38]测量泊松比来定量表征界面的粘结情况。

从国外的研究情况可以看出,无论在双基和NEPE推进剂,还是在HTPB推进剂的贮存性能研究方面已达到非常先进的水平。国外采用光谱学和在推进剂中埋入微型传感器等方法来监测老化,监测技术已达到或接近实用阶段,且这些方法是非破坏性评估方法,有广阔的应用前景。

3 国内固体推进剂贮存老化研究

国内在固体推进剂的贮存老化性能方面做了大量深入细致的研究工作,航天科技集团四院42所、航天科工集团六院46所、西安近代化学研究所、国防科技大学等单位针对推进剂贮存老化性能进行了深入研究,取得了大量的实验数据,对分析固体推进剂失效与预估寿命有一定的促进作用。

衡淑云等[39]对硝酸酯火药进行热加速老化实验,结果发现单基、双基、三基发射药和双基推进剂的安全贮存寿命一般在40年以上,而加入高氯酸铵(AP)、太根(TEGN)等成分改性的双基发射药和推进剂安全贮存寿命大多低于40年。高鸣等[40]对双基推进剂药柱进行气孔率热老化试验研究,探讨了用累积损伤理论和粘弹性分析方法来预测药柱贮存寿命的理论基础和应用价值。张腊莹等[41]采用DMA研究了SJ-1双基推进剂的动态力学性能,表征了其在65 ℃下的老化性能。研究发现,除因结构松弛造成的"物理老化"外,部分增塑剂的逐渐挥发是造成各力学损耗量随时间下降的又一主要原因。范夕萍等[42]利用DMA研究了在65 ℃下老化不同天数的NEPE推进剂的物理性能,确定了物理老化对其力学性能的影响,并以此作为失效的主要模式。

张昊等[43]考察了NEPE推进剂老化过程中结构与力学性能的关系,研究结果表明高温加速老化过程中推进剂样品的抗拉强度和初始模量下降的原因是推进剂粘合剂母体结构的凝胶质量分数、化学交联密度和物理交联密度的下降,NEPE推进剂的降解和解聚由粘合剂母体结构变化引起。张昊等[44]研究了NEPE推进剂力学性能与化学安定性关联老化行为,发现两者的老化行为存在关联性。

贺南昌等[45-46]综述了复合固体推进剂的化学老化机理,并讨论了改善老化性能的技术途径。王春华[47]研究了贮存老化过程中HTPB推进剂凝胶的氧化反应热效应与推进剂力学性能的相关性。张昊等[48]对阿累尼乌斯方程进行了修正,提出了线性活化能法预估固体推进剂贮存寿命,得到了更加准确的预估结果。张昊等[49-50]将静态力学性能参数与凝胶百分数、动态力学性能参数建立关系,实现了HTPB和NEPE推进剂贮存寿命的非破坏性评估;他们[51]还从动力学理论入手,研究了用非破坏性手段预估固体推进剂残留寿命的方法。

李彦丽[52]和张昊等[53]研究了发动机药柱和推进剂方坯老化性能的相关性。赵海泉等[54]研究了应力、湿度、环境-应力联合作用对丁羟推进剂损伤的影响。王鸿范等[55]研究发现丁羟推进剂在定应变条件下贮存时,力学性能大幅下降。鲁国林[56]在对定应变状态下方坯药寿命预测中还发现,在15%定应变贮存条件下,

某 HTPB 推进剂的贮存寿命比非应变状态下缩短了 4 年。张昊等[57]研究了应力应变与固体推进剂使用寿命的关系，研究表明，应力应变的作用等效于降低了推进剂老化的表观活化能，从而加速推进剂的老化，利用推导出的应力应变与动力学公式的关系，预测的推进剂寿命结果更接近于发动机中推进剂的实际使用寿命。

邢耀国等[58]对不同贮存期的某固体火箭发动机所用 HTPB 推进剂药柱进行了大量的力学性能试验，分析了药柱在生产、运输、贮存、勤务处理和点火燃烧等过程的受载状态，使用长期贮存定期检测法预测推进剂药柱的使用寿命。刘德辉等[59]探讨了利用强度和延伸率两种老化数据预估推进剂贮存寿命的方法，并分析了贮存寿命的可靠性。刘兵吉[60]分析了固体推进剂力学性能随温度变化的老化过程，提供了延伸率老化的可靠性模型和可靠寿命的计算方法。阳建红等[61]通过声发射试验检测单轴定速拉伸试验试样的动态损伤破坏过程，指出 HTPB 复合固体推进剂作为高固体颗粒填充的复合材料，材料内部微孔洞、微裂纹的开裂和扩展是其破坏的主要因素。

从国内研究情况可看出，国内在推进剂的贮存性能研究方面虽然取得一定进展，但离国外的贮存性能监测水平方面还有不小差距，需加速推进此方面的工作。

4　结束语

随着固体导弹武器的增加，对固体推进剂贮存性能研究和寿命评估日益重视，固体推进剂贮存老化研究将继续走向深入，根据掌握的动态，今后的发展趋势是：

（1）在与实际贮存条件相近的环境载荷条件下预估。由于固化降温、自身重量、运输振动、贮存循环温度、燃气内压和飞行加速度等因素的影响，推进剂处在受力状态下，与不受力的推进剂方坯药比较，在受力状态下研究推进剂的贮存寿命更具有工程应用价值。

（2）重视老化过程中氧化剂等固体填料与粘合剂界面的“脱湿”研究。粘合剂基体和氧化剂等固体填料的“界面”老化是推进剂应力应变发生变化的重要原因，研究老化过程中的“脱湿”及键合剂对“脱湿”的影响，对改善推进剂贮存后的力学性能具有重要意义。

（3）大力发展老化监测技术。老化监测技术可实时监测发动机中固体推进剂的老化情况，对是否失效和剩余寿命可作出准确判断，对节约经费和加强战备有重要意义。

（4）深入开展固体推进剂老化机理研究。将老化过程中推进剂的微观结构参数与宏观力学性能建立关系，最终建立老化的物理和数学模型，进行数值仿真计算。

（5）使用遗传算法、神经网络、蒙特卡罗和模拟退火等方法进行寿命预测及可靠性计算。如神经网络技术具有高速运算能力，通过网络学习实现非线性函数映射，可以应用于推进剂贮存寿命预测。采用混合算法可以解决单个算法的缺陷，预计混合算法会得到较高的预测精度。

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Review on the Aging of Solid Propellants

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Abstract: The progress of the aging of double-base propellant, nitrate ester polyether (NEPE) propellant, hydroxy-terminated polybutadiene (HTPB) propellants was reviewed. The aging monitoring methods including spectroscopy method and embedded subminiature sensing devices have been used in recent years abroad. Aging under constant strain or stress, interface dewetting, health monitoring system, aging modeling and simulating are the main trend in the future.

Key words: applied chemistry; solid propellant; storage property; aging; review