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不同镀层钕铁硼磁体抗胆汁腐蚀的体外研究 *

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摘要 目的:利用体外浸泡法评价镍镀层、氮化钛镀层和镍 - 氮化钛复合镀层后的钕铁硼磁体抗胆汁腐蚀的效果,为临床磁压榨技术在胆道重建中的应用提供表面改性方案。**方法:**收集临床患者胆汁,采用体外恒温 38℃浸泡磁体,定期称量磁体质量并更换浸泡液,检测浸泡液铁离子浓度。浸泡 30 天后计算各组磁体质量丢失率,扫描电镜观察磁体表面结构。**结果:**胆汁浸泡 30 天后,裸磁体、镍镀层、氮化钛镀层、镍 - 氮化钛复合镀层组磁体质量丢失率分别为 0.57%、0.35%、0.34%、0.19%,不同时间点浸泡液铁离子浓度不同。电镜观察结果显示各组浸泡后磁体表面均出现不同程度的腐蚀斑,镀层出现脱落。**结论:**镍 - 氮化钛复合镀层表面改性处理后的钕铁硼磁体抗腐蚀能力明显优于单纯镍镀层和氮化钛镀层的磁体。

关键词: 钕铁硼;表面改性;磁外科;磁压榨技术;腐蚀;胆汁
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In Vitro Study on the Bile Corrosion Resistance of NdFeB Magnets with Different Coatings*

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ABSTRACT Objective: To evaluate the effect of Nd-Fe-B magnets coated with Ni, TiN and Ni-TiN composite coatings on the bile corrosion through the in vitro immersion method, and provide a surface modification scheme for the clinical application of magnetic compression technique in biliary reconstruction. **Methods:** The bile of clinical patients was collected, and the magnets were immersed at 38 ℃ in vitro. The weight of magnets was periodically weighed and the soaking solution was replaced to measure the iron ion concentration of the soaking solution. After 30 days of immersion, the weight loss rate of each group of magnets were calculated, and the surface structure of magnets were observed by scanning electron microscopy. **Results:** After 30 days of bile soaking, the weight loss rates of magnets of bare magnet, nickel plating, titanium nitride coating and nickel-titanium nitride composite coating were 0.57%, 0.35%, 0.34%, and 0.19%, respectively. The concentration of iron in the soaking solution is different in each time points. Electron microscopy showed that the surface of each group of magnets showed different degrees of corrosion spots after immersion, and the coating appeared to fall off. **Conclusions:** The corrosion resistance of NdFeB magnets after surface modification of nickel-titanium nitride composite coating was significantly better than that of pure nickel coating and titanium nitride coating.

Key words: NdFeB; Surface modified; Magnetic surgery; Magnetic compression technique; Corrode; Bile
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前言

磁外科(Magnetic Surgery, MS)相关技术发展迅速,目前已初步形成了以磁压榨技术 (Magnetic compression technique, MCT)、磁锚定技术(Magnetic anchor technique, MAT)、磁导航技术(Magnetic navigation technique, MNT)、磁悬浮技术(Magnetic levitation technique, MLT)、磁示踪技术 (Magnetic tracer technique, MTT)为核心的应用体系[1]。磁外科的发展虽然只有短短

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的 40 年,但已经历了实验论证阶段、自由探索阶段,现在正处于学科建设阶段。磁压榨技术是磁外科应用最成熟的技术,磁压榨技术中磁体在体内留置时间长、磁体外环境的理化生差异大,在机体不同的使用环境下对磁体的抗腐蚀能力要求不同。因此,探索不同应用环境下磁体的耐腐蚀性能对于推动磁外科的发展具有重要意义。

磁压榨技术(MCT)已目前涉及到胃肠道吻合重建[2-6]、胆肠吻合[7-10]、食管闭锁重建[11-14]、直肠阴道瘘闭合修补[15-18]、血管吻合[19-24]、消化道造瘘[25-28]。在不同的应用目的下,磁体所处的外环境特点、留置时间差异性较大,对磁体的表面改性要求也提出了不同的要求。因此,探索胆汁对不同镀层后的磁体的腐蚀行为,对推动磁压榨技术在胆道重建方面的有重要意义,将为未来规范磁体设计、建立磁体体内植入标准提供重要实验数据支持。

本研究以临床胆道磁压榨重建手术为背景,设计体外胆汁浸泡实验,通过测量和计算质量丢失率、浸泡液铁离子浓度检测及扫描电镜观察比较了镍镀层、氮化钛镀层、镍 - 氮化钛复合镀层钕铁硼磁体的抗胆汁腐蚀性能。

1 材料与方法

1.1 材料

烧结型 N45 钕铁硼永磁体(购于陕西金山电器有限责任公司),人体胆汁(取自临床行 PTBD、ENBD 及 T 管引流的患者),电热恒温振荡水槽(上海一恒科技有限公司,DKZ 系列)、电子天平(赛多利斯科学仪器北京有限公司,BSA323S)、全自动生化分析仪 (深圳雷杜生命科技,Chemray 240)、酶标检测仪(BioTeK,Epoch)、扫描电镜(日本 HITACH 公司,TM-1000)。

1.2 钕铁硼磁体样品制备及分组

以本团队现有的不同镀层的钕铁硼磁体(由陕西金山电器有限责任公司加工制备)共 40 个作为检测对象,其中包括:镍镀层磁体(n=10)、氮化钛镀层磁体(n=10)、镍 - 氮化钛复合镀层磁体(n=10),另取裸磁铁(n=10)为对照组。

取 15 mL 离心管,其内装入 10 mL 临床收集的人源胆汁,所有磁体称重后每个离心管内置入一个磁体样品。将所有盛有胆汁和磁体的离心管置入 38℃ 恒温振荡水浴箱内。

为避免胆汁理化生特性发生改变,胆汁每 3 天更换一次,每次更换胆汁时先抽取 1 mL 浸泡液 -80℃ 留存。操作流程详见图 1。

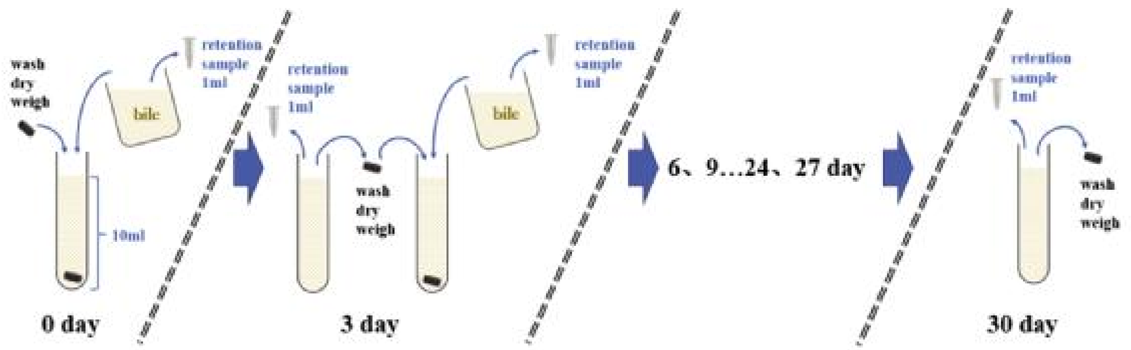

图 1 胆汁浸泡磁体实验流程示意图

Fig. 1 Schematic diagram of experimental procedure for bile immersion magnets

1.3 磁体质量

每次更换浸泡液时称重磁体质量,取出的磁体采用机械法去除磁体表面腐蚀颗粒及沉淀附着物,在流动水下用软毛刷清洗磁体表面,去除磁体表面杂质,然后干燥箱内干燥 30 分钟后称重并记录。获取磁体浸泡前及胆汁浸泡后每三天的质量,绘制质量变化曲线;按下述公式计算磁体质量丢失率,磁体质量丢失率 =(磁体初始质量 - 磁体浸泡 30 天后的质量)/ 磁体初始质量× 100%。

1.4 浸泡液铁离子浓度检测

每次更换胆汁时,留取胆汁原液及每管浸泡液胆汁 1 mL 冻存备用,自动生化检测仪检测浸泡液内铁离子浓度。

1.5 扫描电镜观察

所有磁体浸泡 30 天后取出,流动水下软毛刷清理表面附着物,超声波清洗仪清洗磁体,恒温干燥箱干燥磁体。电镜观察磁体表面腐蚀情况。

1.6 统计学分析

磁体质量及浸泡液铁离子浓度均以均数± 标准差的形式表达,磁体质量丢失率以百分数形式表达,应用 SPSS 19.0 软件对数据进行统计分析,*P*<0.05 为有统计学意义。

2 结果

2.1 各组磁体质量变化情况

每三天称量一次各组磁体质量,绘制质量变化曲线(如图 2),裸磁体在浸泡期间基本成下降趋势,而且随着浸泡时间的延长,下降趋势越来越明显;镍镀层早期质量较稳定,随后呈现梯度下降趋势,随时间延长下降曲率越来越大;氮化钛镀层和裸磁体在早期质量明显下降,但不同于裸磁体的是质量下降过程中存在几个平台期(3-9d、12-21d、24-30d),说明具有一定的稳定性;镍 - 氮化钛复合镀层组磁体在早期(0-12d)质量稳定,说明早期就具备良好的抗腐蚀能力,和氮化钛镀层类似在随后也出现了平台期(15-21d、24-30d),说明在长期浸泡中抗腐蚀能力较稳定。各组磁体胆汁浸泡前后质量减少情况见表 1。

2.2 浸泡液铁离子浓度

检测留取的胆汁原液及浸泡完磁体后的胆汁铁离子浓度,绘制变化曲线如图 3 所示。各组磁体浸泡完的胆汁铁离子浓度在不同时间点均不同,提示磁体腐蚀与时间并非成线性关系。

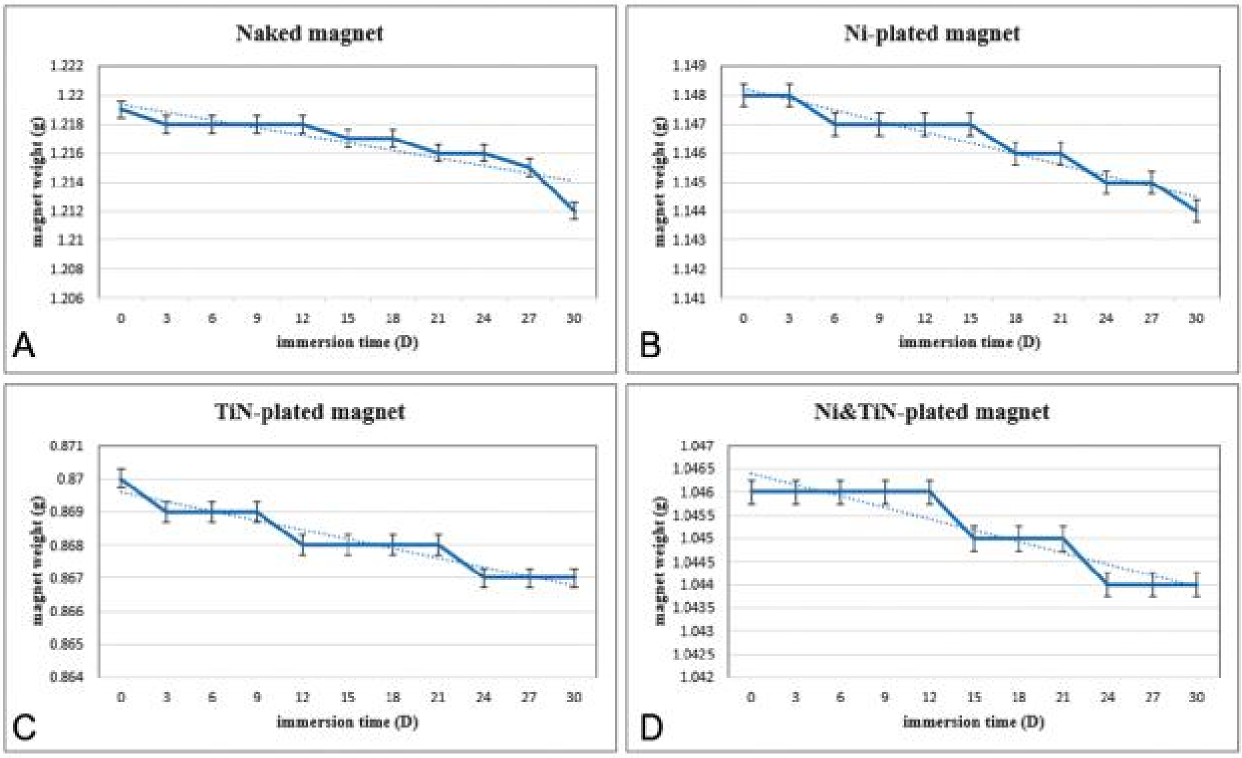

图 2 各组磁体不同时间点的质量

Fig. 2 The quality of magnets at different time points in each group

表 1 各组磁体浸泡前后质量差异

Table 1 The Weight change of magnets after immersion in different groups

Surface modified	Initial weight(g)	Final weight(g)	Weight loss rate (%)	*P* value
Naked magnet	1.219± 0.039	1.212± 0.037	0.57	
Ni-plated magnet	1.148± 0.030	1.144± 0.068	0.35	$P<0.05$
TiN-plated magnet	0.870± 0.009	0.867± 0.009	0.34	$P<0.05$
Ni&TiN-plated magnet	1.046± 0.009	1.044± 0.009	0.19	P<0.05

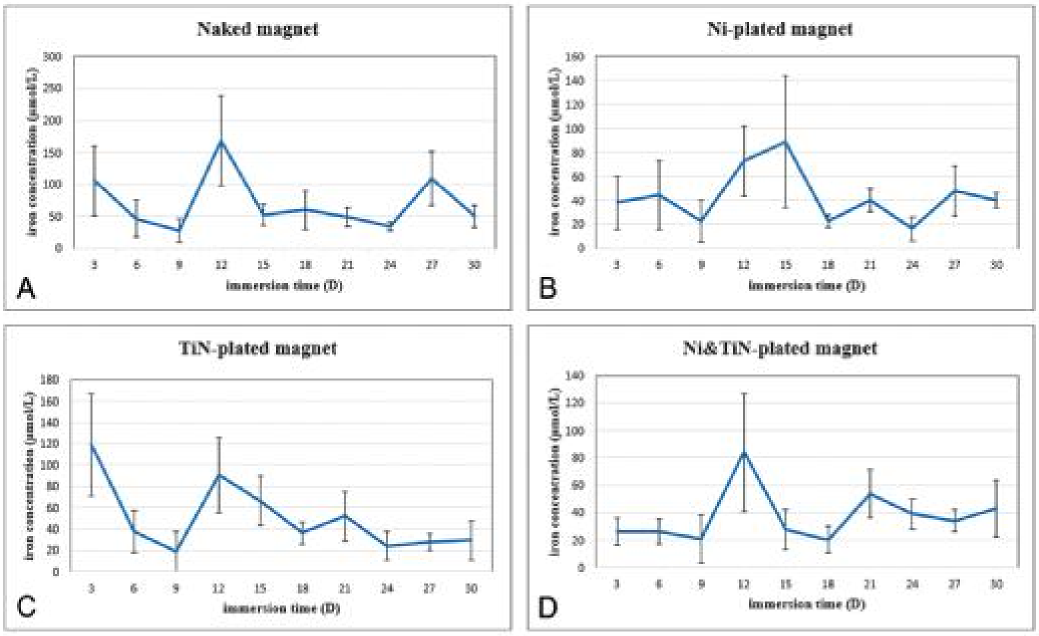

图 3 浸泡液铁离子浓度变化曲线

Fig. 3 The variation curve of iron ion concentration in soaking solution

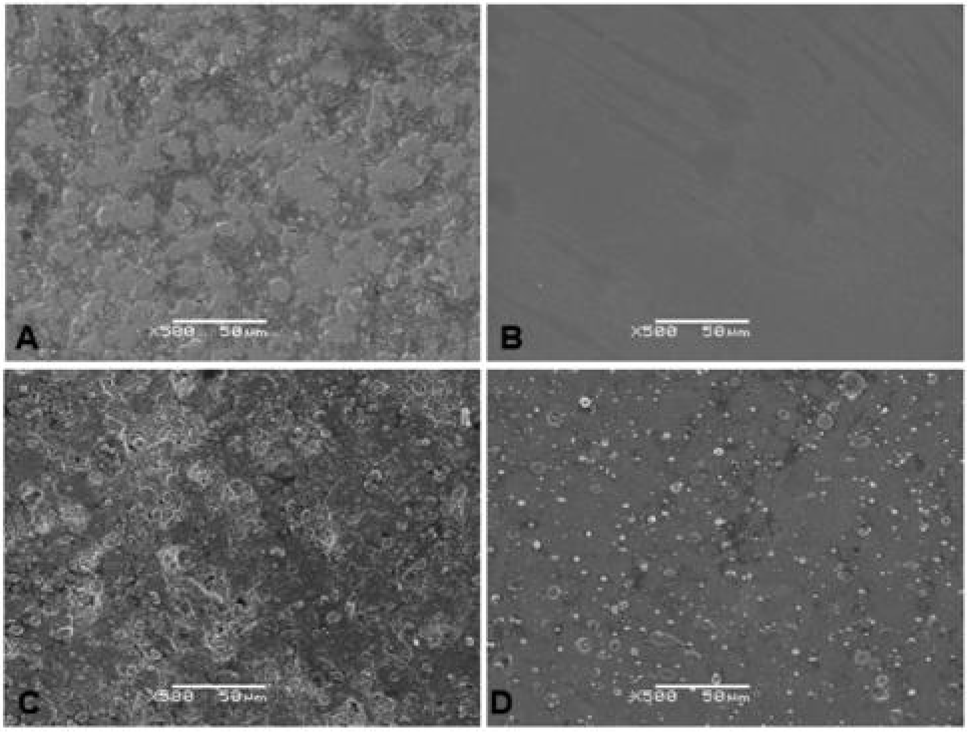

图 4 浸泡前各组磁体扫描电镜

Fig. 4 Scanning electron microscopy of magnets in each group before immersion

(A:裸磁体;B:镍镀磁体;C:氮化钛镀层磁体;D:镍 - 氮化钛复合镀层磁体)

(A: uncoated magnet; B: nickel coated magnet; C: titanium nitride coated magnet; D: nickle-titanium nitride coated magnet)

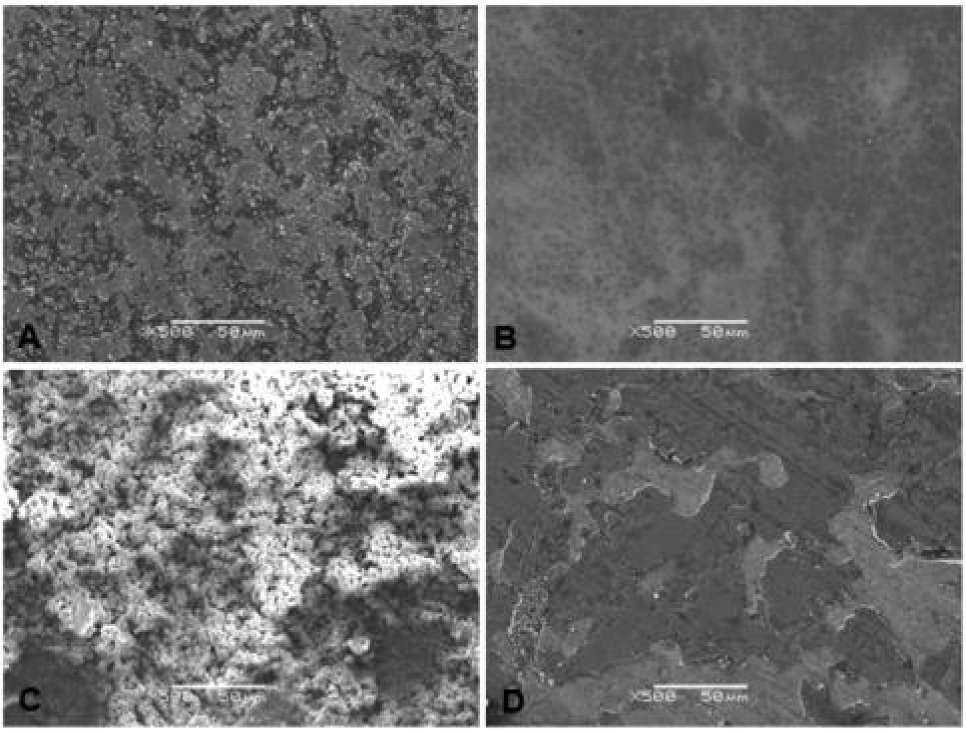

图 5 浸泡后各组磁体扫描电镜

Fig. 5 Scanning electron microscope of each group of magnets after immersion

(A:裸磁体;B:镍镀磁体;C:氮化钛镀层磁体;D:镍 - 氮化钛复合镀层磁体)

(A: uncoated magnet; B: nickel coated magnet; C: titanium nitride coated magnet; D: nickle-titanium nitride coated magnet)

但各组胆汁不同时间点铁离子浓度变化曲线显示在 12 天左右为铁离子浓度高峰时期，提示在 12 天时各组磁体受胆汁腐蚀程度较大。

2.3 扫描电镜表面观察情况

浸泡前各组磁体扫描电镜观察见图 4。裸磁体表面呈现出凹凸不平和氧化腐蚀点，而镍镀层表面则为非常光滑平整的表

面,氮化钛镀层表面为类似微小颗粒状面貌,镍 - 氮化钛复合镀层表面较为平整,但可见大量空泡样结构在磁体表面。

胆汁浸泡 30 天后的各组磁体扫描电镜观察见图 5。裸磁体表面凹凸不平加深,可见明显腐蚀点,镍镀层磁体表面可见镀层脱落并形成明显腐蚀斑,氮化钛镀层磁体表面出现明显腐蚀斑块形成的凹陷,镍 - 氮化钛镀层磁体表面出现镀层的片状剥脱,剥脱面深层仍可见镀层覆盖,未见明显的腐蚀点。

3 讨论

钕铁硼最初在医学中主要被用于口腔正畸,因此,有关不同镀膜处理后的钕铁硼磁体抗唾液腐蚀的研究较多 [29]。近些年,在磁外科的快速发展下,钕铁硼磁体被广泛用于消化道吻合重建中, 而消化道的理化生环境与口腔环境差异非常大,因此探讨在消化道环境下不同镀膜的钕铁硼磁体抗腐蚀能力对于磁外科的发展及建立钕铁硼医疗器械体内可植入标准业均具有重要意义。

电镀镍具有成本低、工艺要求容易实现等优点,是工业中最常见的钕铁硼表面防腐处理方案, 而且在大部分情况下,电镀镍能够满足常规表面防腐要求。氮化钛属于非金属氧化陶瓷,化学性能稳定,耐腐蚀性能强,且有研究显示氮化钛镀层具备更好的生物相容性[30],且目前工艺成熟、常用的表面改性方法之一。本团队在前期临床研究中,通过对 41 例磁压榨胆肠吻合患者的统计发现胆肠吻合口形成的平均时间为 19 天(14.5-23 天)[31],因此本研究中体外浸泡时限设定为 30 天,能够满足对临床研究需要。本研究结果显示在体外浸泡第 3 天和第 12 天时,浸泡液铁离子浓度出现小高峰,提示磁体在这两个时间点腐蚀行为较重。而质量改变曲线可见镍 - 氮化钛复合镀层的磁体在 12 天以前质量基本保持稳定, 其早期抗腐蚀能力明显优于镍镀层和氮化钛镀层组的磁体。磁压榨技术在实现吻合时,受压组织发生缺血 - 坏死 - 脱落,而压榨旁组织则发生粘连-修复 - 愈合的病理变化[32],因此在吻合口形成之前,磁体应保持稳定的压榨力, 而稳定压榨力的维持则需要良好的抗腐蚀能力。本研究结果提示镍 - 氮化钛复合镀层的磁体在吻合口形成前能够很好地保护磁体免于腐蚀。

综上所述,镍 - 氮化钛复合镀层能够显著提高钕铁硼磁体抗胆汁腐蚀能力,能够满足胆道重建手术中对磁体的表面防护要求。

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