

机电学院

053 系

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ON-MACHINE MEASUREMENT AND ERROR COMPENSATION FOR TOUCH-TRIGGER PROBES

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ABSTRACT

This paper advances a method to implement the on-machine measurement (OMM) with the touch-trigger probe, also called switching probes. Some of the advantages and disadvantages for touch-trigger probe are discussed. However, the touch-trigger probe errors exist and become one of the major errors for the measurement accuracy. Major factors that influence the probe measurement have been analyzed. The basic technique of probe measurement error modeling with artificial neural network was researched, and also the probe measurement error compensation with 3-layered back-propagation artificial neural network was presented. At last in the experimental system composed of DIXI 50 machining center, Fanuc 16i control system,

Blum CNC P82.046 probe and PC, valid the correlated techniques. In addition, the connection and communication between the machining center equipped with probe system and the computer have been introduced. The experiment indicated that, using the touch-trigger probe makes on-machine measurement more automatic and efficient. And by using the back-propagation neural network for error compensation make on-machine measurement more precise.

KEY WORDS: On-machine measurement, Error compensation, Touch-trigger probe, Machining center

1. Introduction

Today there are two measurement methods for conventional manufacturing process according to the way whether the machining process and inspection process are done on the same equipment [1]. One is offline measurement that part inspection is done with stand-alone measurement instruments such as coordinate measuring machines (CMM), which are generally located at a separate room apart from a machine center [2]. And the other is online measurement, also called on-machine measurement (OMM), that machining process and inspection process are both on machine center.

inevitably reduces the measurement precision due to the secondary fixture error when transfer the workpiece from machine center to CMM.

To overcome these problems, the methodology of OMM is now widely accepted. As for OMM, it also has two types. One is in-process gauging, and the other is in-cycle gauging[5]. In-process gauging means machining and gauging at the same time. The in-cycle gauging means gauging is done only before or after the machining process, however the workpiece will not be disassembled from fixture. In this paper in-cycle gauging will be mainly discussed.

The offline measurement method by CMM is the most popular now. Many research works are done about measurement by CMM [2-4]. However this increases the overall manufacturing cost and time to obtain the final product, and the bottleneck phenomenon may be caused by the product stagnation due to the time lag between the machining and inspection process. In addition it is hard to measure the complex, large-sized parts [2]. Furthermore, it

An on-machine measurement (OMM) system as illustrated in Fig. 1 is implemented using a commercial touch probe (CNC P82.046 probe, Blum inc), also called switching probes. There are some advantages to use touch probe in an OMM system. First a touch probe is a relatively inexpensive and easy-to-use. In a machine center, the motion of a probe treated as

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a cutting tool, is controlled by G code or macro program. After the machining process is finished, the touch probe replaced with a cutting tool starts the measurement in the normal direction to the machined surface. Second as an accessory of certain machine center the probe is easy to integrated with CNC controller system. The probe system is composed of optical module communication is used to transmit the measurement program to the CNC controller and receive the measured data for further analysis using a personal computer. So OMM system with touch probe can reduce the

probe (OMP) and optical machine interface (OMI). OMP, located between the probe head and the shank, receives machine control signals and transmits probe signals. Communication between the probe and the OMI is done via the optical transmission system. RS-232 serial

production time and cost, can be widely used for automating and speeding part processing, eliminate errors caused by secondary fixture.

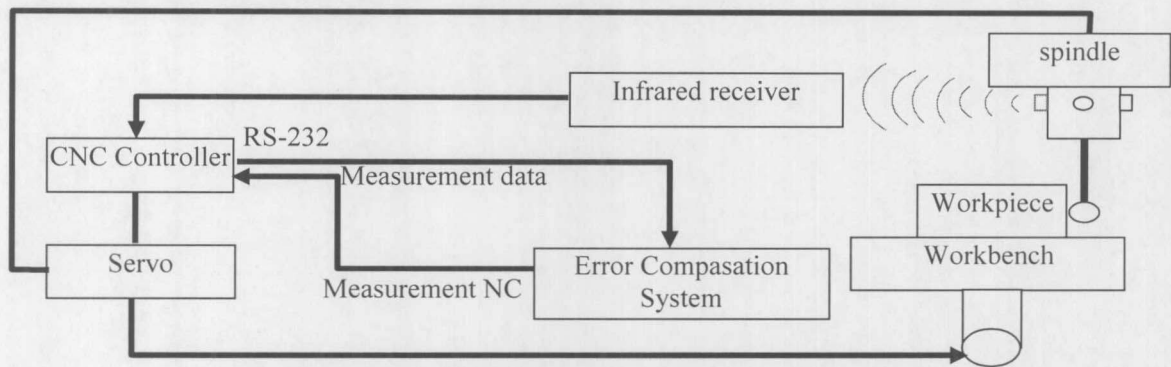


Fig. 1 An on-machine measurement (OMM) system structure

However for high-accuracy applications with touch probe, it measures parts along the machine tool axes, the measured data inevitably include the probing errors originated from the structure of a touch probe, and the positioning errors originated from the inaccurate axis motion of a machine tool. There are largely systematic errors influence the accuracy, such as pre-travel variation in different probing directions, stylus length, diameter, measuring speed, trigger force, etc. [6-7]. These errors should be eliminated from the measured data to obtain the true machining error.

probe measurement error modeling with artificial neural network was researched, and also the probe measurement error compensation with 3-layered back-propagation neural network was presented. At last in the experimental system composed of DIXI 50 machining center, Fanuc 16i control system, Blum CNC P82.046 probe and PC, valid the correlated techniques. In addition, the connection and communication between the machining center equipped with probe system and the computer have been introduced. The experiment indicated that, using the touch-trigger probe makes on-machine measurement more automatic and efficient. And by using the back-propagation neural network for error compensation make on-machine measurement more precise.

This paper advances a method to implement the on-machine measurement (OMM) with the touch-trigger probe. The basic technique of

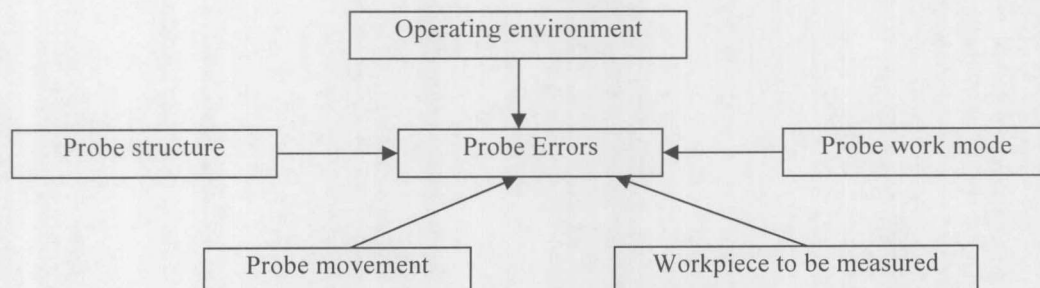


Fig.2 The probe error model

2. The major errors that influence the probe precision

A generalized probe error model is discussed in paper [8], in which the influencing factors of probe are group into the following four categories: impact force, probe rigidity, stylus rigidity and operating environment. Besides these factors we also find there some other factors, such as the probe movement, the structure of probe, the work mode of probe. So the probe error model can be rearranged as Fig.2.

In Fig.2, there are five categories that consist of several factors respectively. Operating environment includes the environment temperature and machining temperature. Probe structure includes probe rigidity, stylus rigidity, stylus ball's radius, stylus length and single emitting angle. Probe movement includes

feedrate, direction of probe to approach workpiece and impact force. Workpiece to be measured includes the shape, material of workpiece. Probe work mode includes measurement path and the number of point to measurement.

Each factor could impact the measurement precision more or less. For example, we know that high probe approach speed results in bigger forces transmitted through the probe system and so a bigger distortion of the stylus. As far as feedrate of probe concerned, as show in Fig.3, at the measuring time, the measuring axis should move with constant federate (Fig.3 (a)). Measuring when the machine is acceleration (Fig.3 (b)) or deceleration (Fig.3 (c)) can decline the repeatability or result in measurement error. And so calibration and tool measurement should be done with the same federate.

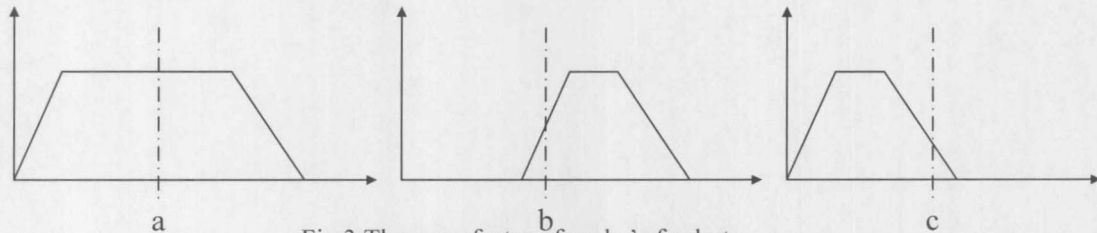


Fig.3 The error factor of probe's feedrate

3. Error compensation with back-propagation neural network

All the factors mentioned above may influence measurement precision, it's hard to set up a model to analyze how much each factor the combination of each factor contribute to errors.

One practical method of formulating such a complicated model is to use back-propagation artificial neural networks (BP-ANN). It has been found useful in high precision measurement, and is particularly suitable for probe error modeling because of the complexity involved.

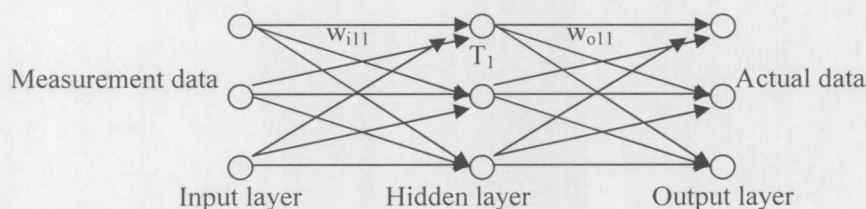


Fig.4 BP-ANN model for error compensation

A 3-layered BP-ANN mode for probe error compensation is depicted in Fig. 4. The neurons in the input layer represent the measurement data, and in the output layer the actual data. The essential of ANN is using the training samples (measurement data), to train the BP-ANN, to get the best parameters, so as to map measurement data to actual data. The best parameters are stored in the BP-ANN in the form of weights, which represent the tight degree of different layer, as w_{i11} means the tight degree between input layer's first neuron and hidden layer's first neuron, and w_{o11} means the tight degree between

hidden layer's first neuron and output layer's first neuron. T_1 is the threshold of first neuron.

As measuring, the probe touch certain point of a workpiece in the machining center, and get the position of this point, that is measurement data. However, due to the existence of probe errors, this measurement data is incorrect, and there are several methods to get the correct data. So we need train enough measurement data using BP-ANN to map them to correct data. It's important to get the neurons in hidden layer

because the input and output layer are already known. But it's hard to get the neurons in hidden layer, the only way to get them is to train, test and adjust. When test the model, new points should be used instead of the samples.

The choice of samples is arbitrary and is better to cover the whole workpiece. Once the model has been trained and tested, it can be implemented either on-line or off-line to compensate for probe errors.

4. Case study

The on-machine measurement (OMM) system structure is shown in Fig.1. The system hardware is composed of DIXI 50 machining center, Fanuc 16i control system, Blum CNC P82.046 probe, micrometer, standardized ring gauge and PC. The probe styli is made of steel and carbon with $\phi 5\text{mm}$, length 50mm. The diameter of standardized ring gauge is $\phi 10 + 0.0012\text{ mm}$.

In this case, by using high precise micrometer, the standardized ring gauge is put on the MC, whose center is consistent with the center of spindle, as shown in Fig.5. Then make this center point as origin of workpiece

coordinate system, so each point in the standardized ring gauge in the direction of +X, -X, +Y, -Y can be pinpointed by calculating, also called theory points. These points can be treated as points in output layer. And the points measured by probe are points in input layer, also called actual points. By this way, the margin expresses the error of the whole on-machine measurement system. In addition, the points in the direction of +X, -X, +Y, -Y are symmetry, so we only measure the points in the direction of +X. Table1 shows the training sample data.

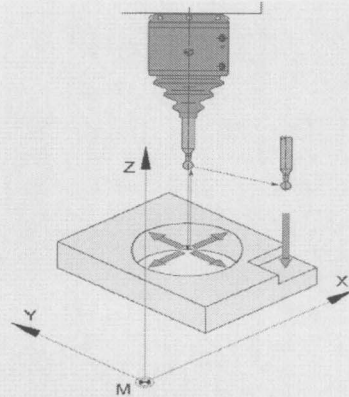


Fig.5 standardized ring gauge

Table1 The training sample data in +X

	actual points		theory points			actual points		theory points	
	+X	Y	+X	Y		+X	Y	+X	Y
1	0.0000	10.0012	0.0000	10.0000	11	10.0009	0.0000	10.0000	0.0000
2	1.5650	9.8792	1.5636	9.8770	12	9.8754	-1.5588	9.8782	-1.5557
3	3.0899	9.5133	3.0887	9.5111	13	9.5104	-3.0789	9.5135	-3.0811
4	4.5302	8.9140	4.5278	8.9111	14	8.9118	-4.5287	8.9148	-4.5307
5	5.8723	8.0961	5.8753	8.0920	15	8.0939	-5.8667	8.0967	-5.8688
6	7.0712	7.0698	7.0683	7.0739	16	7.0768	-7.0653	7.0795	-7.0626
7	8.0839	5.8788	8.0874	5.8817	17	5.8847	-8.0859	5.8881	-8.0827
8	8.9035	4.5416	8.9075	4.5449	18	4.5501	-8.9060	4.5519	-8.9039
9	9.5101	3.0927	9.5086	3.0962	19	3.1065	-9.5033	3.1038	-9.5061
10	9.8719	1.5695	9.8758	1.5714	20	1.5815	-9.8721	1.5793	-9.8745

There are tow neurons in input layer express +X and y of actual points, and two neurons in output layer express +X and y of theory points. Then Using BP-ANN to train the sample data and the net learn from training. The weight of BP-ANN, including weight between input and hidden layer and weight between hidden and output layer, may use a random number between -1 to 1. The weight and

threshold is not a constant, the change rule is defined like formula (1). Here, β means learning speed used to adjust learning convergence speed. To quicken convergence, β could exceed 1, otherwise less than 1. And $\square x_n$ stands for the difference between input x_{n+1} and x_n . In this case the value of β is 0.75 and 1.33.

$$\begin{cases} w(n+1) = w(n) + \beta \square x_n \\ T(n+1) = T(n) + \beta \end{cases} \quad (1)$$

The maximal training time is 10000, and sum-squared error (SSE) is 0.001. The key

problem lies in the exact quantity of hidden layer's neurons. We first use one neuron in

hidden layer to train the net. In the training process, when in the set time and the SSE is less than 0.001, we think the training process finished and save the weight, threshold and quantity of hidden layer's neurons in a text file. And this process is so called error compensation.

Afterward, measure another data to test the net that has been trained. If the result can't be accepted, then the BP-ANN will be trained again, otherwise it's a good training. Table 2 lists the test result.

Table2 The error compensation data in +X

	actual points		theory points		compensation points	
	+X	Y	+X	Y	+X	Y
1	2.0744	9.7853	2.0780	9.7817	2.0753	9.7848
2	4.0681	9.1404	4.0654	9.1363	4.0670	9.1399
3	7.4312	6.6987	7.4285	6.6946	7.4301	6.6978
4	8.6608	5.0103	8.6575	5.0047	8.6589	5.0091
5	9.9407	1.0562	9.9444	1.0529	9.9428	1.0560
6	9.9499	-1.0321	9.9461	-1.0366	9.9477	-1.0331
7	8.6623	-4.9951	8.6657	-4.9905	8.6632	-4.9939
8	7.4431	-6.6865	7.4395	-6.6823	7.4411	-6.6852
9	4.0838	-9.1244	4.0804	-9.1297	4.0819	-9.1261
10	2.0976	-9.7740	2.0941	-9.7782	2.0957	-9.7753

In this case, we all get 120 sample points in four directions, including 80 data for training and 40 data for test. All these 120 sample points are sent to the PC from machine center by RS232, and could be stored in the database for later net training and testing. The transfer marco in FANUC control system is POPEN and PCLOS which are originally used to send data to a series printer. And we write a program to watch the series port and get data once the data is sent from machine center.

5. Conclusion

A method using the back-propagation artificial neural network (BP-ANN) for error compensation has been advanced after analyzing the major errors that influence the probe precision. An on-line measurement system with Blum CNC P82.046 touch trigger probe has also

The method mentioned above can also be used to handle the direction of $-X$, $+Y$, $-Y$, and is similar with $+X$. Due to the structure of the probe, and the characteristic of machine center, the error in direction Z that is the spindle, can be ignored compared the error in other directions. So in this case we didn't consider the error of direction Z. The data listed in table2 show that the method for error compensation of touch-trigger probes is effective, and direction $+X$ is better trained than direction Y.

6. Acknowledgement

Thanks for prof. Ye wenhua, he gave me such a chance to complete the research. And also thanks for the China Electronics Technology

been introduced. From a case mentioned above, it validates the measurement system with touch trigger probe and the method using BP-ANN for error compensation. In addition, the method can be used off-line, only if there is a file in which all the data has correct format that could be read by the measurement system.

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“机电一体化”课程教学实践与探索

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摘要:“机电一体化”课程教学在教学内容上突出系统总体设计理论、方法,以及机电结合的设计思想,在教学过程中使用多个完整的工程实例贯彻始终来加强学生的印象。在注重课堂教学的同时,加强现场示范课、实验课和实例设计来培养学生的工程实践能力和创新能力。

关键词:机电一体化;创新能力;工程实践

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一、“机电一体化”课程教改思路

“机电一体化”课程作为机械工程及自动化专业的一门院定选修课,已经在该专业开展多年。该课程是机械设计、数控技术、微机原理等课程的综合,并在此基础上更加注重以当前比较前沿的机电控制技术为内容,结合具体的工程案例与实验环节进行教学。从中也可以看出机电一体化课程符合创新教育的基本过程,即基本能力培养,实践能力训练和综合知识应用^[1]。

目前随着生产自动化的要求越来越高,社会对具有机电一体化知识的学生也比较青睐。选修该课程的学生相对较多,也对该课程提出了更高的要求。因此本课程结合教改项目“信息化制造仿真实验策略研究与实验系统开发”,从开展素质教育出发,以培养学生的创新能力和工程实践能力为目的,适应当前社会对人才素质教育培养的要求,我们对该课程的教学内容、教学方法以及教学实验进行了探索和实践,图1就是本课程的教改思路。第一就是课程内容的更新。在学生掌握基本理论的基础上,本课程必须能跟踪新技术的发展和最新应用,并利用

工程实例来说明各个知识点的综合应用。第二,就是对学生实践环节的要求。机电一体化课程由于其自身的特点,必须使学生有现场观摩和自己动手的机会,这样才能使学生对所学有深刻的印象。同时,由于机电一体化课程是一门大综合的课程,是学生前面所学知识的综合应用,所以该课程对全面培养学生的创新能力和综合素质非常适合^[2]。而且生活中到处存在机电一体化产品或有可能使用机电一体化产品的地方,学生有许多想象空间和灵感可以发挥,必须给学生提供发挥主动性的机会。

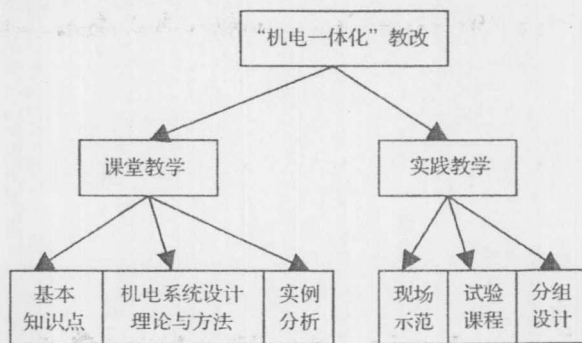


图1 机电一体化教改思路

由于本课程涉及面广,而学时数较少,因此在

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讲授课程中我们贯彻“少而精”和“开放式,研究式”的学习原则,用较少的课时,较全面、系统地讲授机电一体化技术的基本含义、方法和应用,使学生能够掌握本门课程的基本概念,方法和技能,并具有初步的机电一体化分析、设计和应用能力。

二、课程教学内容与方法的改革

“机电一体化”是机械工程及自动化专业四年制本科的选修课,是一门综合性较强的课程,具有重要的工程应用意义。本课程的全新知识点比较少,其重点在于将学生三年来所学的各种知识进行综合的应用。它综合了自动化、机械制造、传感器、计算机和电子线路等众多知识点^[3]。所以,本课程既有理论性,又有较强的综合性和实践性。从教学大纲上看,机电一体化课程的教学内容与部分其他课程如数控技术、机械设计、传感器技术等课程存在相当的重复部分,因此本课程最大的教学难点在于学生对本课程的许多知识点已经学过但又未掌握或实际应用。所以首先从教学内容上就不能让学生觉得作为选修课的机电一体化仅仅是上述课程的重复,其次要使用更多的完整的工程实例来让学生知道所学知识的可以在什么场合使用以及如何使用,为此必须对机电一体化教学内容有正确而又周到的安排。

同时本课程的难点也表现了本课程的教学特色,即根据学生前面所学相关知识教授这些知识点的实际应用情况,特别是突出机械与电气相结合的设计方法与应用。通过实例教学可以极大地提高学生对所学理论知识的理解和应用,开阔学生对当前机电系统发展的认识水平。再结合一些实际的工程项目,更有利于提高教学效率和培养学生的兴趣。

1. 优化教学内容,提高课堂教学质量

在课堂教学的内容安排上,采用了基础知识的复习在先,专业理论学习在后,同时结合具体工程实例的顺序。因为学生对相关专业基础知识淡忘了,让学生完全依靠自己进行复习不切实际,而过于笼统地复习作用又不大,学生反而会无所适从。尽管复习基础理论知识损失了一定的课时,但我们认为是值得的。从教学效果出发,不能完全寄希望于学生的自主性。特别是与传感器、电机、单片机、PLC的内容,尽管教科书上也讲了许多内容,但更多的是原理性的,与许多课程重复。所以教学中我们认为有必要对其进行梳理,主要讲述这些知识点的应用实例。教师将总结后的基础知识系统地给学生复习一次后,既减少了学生负担,也提高了学习效率。为

了在短时间内回顾以往的知识点,目前采用了幻灯片和动画短片的方法表达重要的知识点,既形象直观,促进思考,又达到了辅助的教学目的。在使用多媒体教学过程中,我们也注意到了每个知识点都给学生留半分钟到3分钟的时间,保证学生有思考和发问的时间。

比如我们在讲解机电一体化系统设计方案的评价方法时,我们使用了评价函数法,这种方法在学生以前的学习中已经学过。我们首先简单回顾了评价函数法的计算过程,然后通过使用该方法对一个电子秤总体设计方案进行评价,以电子秤的电机、送料器、传感器、放大器和A/D转换模块等硬件的实现方式、精度、寿命及成本四方面进行评价,使学生对评价函数法从理论和实践中获得了深刻的理解。

2. 实例教学,培养综合分析能力

由于“机电一体化”课程的实践性很强,理论教学过程中必须与工程案例相结合才能达到预期目标。使用案例教学,可以使学生对知识的理解从具体到抽象,从个别到一般,这符合人类认识的进规律^[4]。案例多一点,理论简化一点,对于刚接触这门课程的学生来说是很重要的,尤其许多学生就是看中这门课程的实用性。一方面可以极大的提高学生们的学习本门课程的兴趣,扩大他们的眼界。另一方面可以间接培养学生们的理论知识与工程实践相结合的观点。本课程结合“信息化制造仿真实验策略研究与实验系统开发”的开发过程,重点向学生们介绍了本项目中完成的“柔性自动化物料储运教学实验系统”、“柔性化自动化装配教学模拟实验系统”、“笛卡儿式教学机械手”及多个科研项目中完成的工程实例。

同时通过实例讲解,可以使学生认识到机电结合的设计理论与方法,及与传统机械方法的区别和优点。比如,我们在讲解电子调速器时,就首先讲解了以前机械方式的调速器,即通过转速变化引起离心力的改变,进而利用杠杆原理改变阀门的开度而反方向地改变转速。而电子调速器则依靠传感器和PID控制器来调节转速变化,使之趋向于设定转速。由于目前电子产品的模块化、高精度及可编程,电子调速器对速度的调节更精确,而且实现与维护、功能扩展也更方便。

3. 组建综合实验教学平台,增加学生的动手与实践机会

学生要有现场观摩与自己动手实践的机会,即必须安排专门的现场课与实验课程。任何知识不经过自己的摸索,都不会有深刻的印象。在实践环节

上,有现场示范课,让学生了解机电设备的设计、制造、装配和运行以及各种传感器、气动和液压元件、各种电机的运用,并进行现场讲解。结合“信息化制造仿真实验策略研究与实验系统开发”教改项目的成果,本课程建立了教学网站,网站上在重点介绍了典型机电一体化元件,如同步系统、气动元件、传感器、PLC等。还制作了多个实验系统的装配过程和运行过程动画。还有就是实验课。在本课程之后安排了总共10课时的“机电一体化系统机械机构创意组合实验”、“机电一体化控制实验”及“PLC与上位机通信实验”。

通过现场示范课和实验课,学生们对机电一体化系统有了更深入的认识。比如,以前对PLC、变频器等的认识都停留在书本上,现场示范课真正知道了PLC、变频器以及如何对它们进行编程、接线及设置等。在多个实验系统的演示中,学生们认识到在考虑抗干扰的情况下如何结合机械结构对强电路、控制线路的走线、布线等,将系统设计的又实用又美观。

为进一步开拓学生的思路,在课程的考核中,成绩的一个重要组成部分就是将三到四名分为一组,根据其生活体会和学习心得,设计一个用于日常生活的机电一体化系统,每个学生分别设计机械部分、采集与控制部分、动力及执行部分。同时我们在

课上将专门使用两课时讲解其中比较有意义的设计。这样的实践形式极大提高了学生的学习兴趣 and 主动性。

三、课程改革的实践效果

从机电一体化课程改革的效果看是比较明显的。教改项目获得2005年校优秀教学成果一等奖。该专业的学生选修本课程的人数基本是百分之百的。利用多个实验系统,使学生加深对机电一体化技术、先进制造技术有了深入了解,从本来比较抽象的理论表述变成一个可以进行实际操作的实验系统,哪怕是现场示范课中的一次综合性演示实验,许多学生都表示有很大的收获。

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Research and Practice of Teaching Mechatronics

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Abstract: The teaching of mechatronics on the one hand must enhance the overall design theory and method of mechatronics system, especially the integration of mechanics and electrics. On the other hand, several perfect engineering cases must be used throughout the whole course to impress the students. Besides, paying attention to classroom education, on-the-spot teaching, experiments and engineering design must be emphasized to train the ability of technological creation and the ability of engineering practice.

Key words: mechatronics; ability of technological creation; engineering practice

冻干机控制系统实现及工艺参数优化

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摘要: 真空冷冻干燥是一种使物料在低温低压下脱水的干燥工艺, 具有许多优点。本文以某冻干机为研究对象, 设计与开发了其控制系统并进行了相关实验。利用二次正交分析法对实验结果进行了分析, 对冻干的关键参数时间、温度和真空度进行了优化。实验表明优化的参数可行。

关键词: 冷冻干燥; 控制系统; 工艺参数; 优化

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Realizing control system of freeze-drying machine and optimising on technical parameters

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Abstract: Vacuum freeze-drying is a technical that make a material dehydrate in low temperature and low pressure, and it has many merits. A control system is designed and developed based on a certain freeze-drying machine. According to the test result, by the method of quadratic orthogonal experiment, the key parameters including time, temperature and vacuum degree of freeze-drying are analysed and optimised. The test proves the optimised parameters valid.

Key words: freeze-drying, control system, technical parameters, optimise

Qian XM and Lou PH. Realizing control system of freeze-drying machine and optimising on technical parameters. Computers and Applied Chemistry, 2006, 23(11):1102-1104.

1 引言

真空冷冻干燥(冻干)是一种使物料在低温低压下脱水的干燥工艺^[1]。与其他干燥方法相比,冷冻干燥具有许多优点^[2],如可避免物料的热分解和氧化;可极好地保持物料原有的色、香、味、形和营养价值;保持物料生化和药理特性;易于分切、高复水性、储藏性能优良等。然而电耗高、干燥时间长是冷冻干燥的缺点。如何缩短干燥时间,提高生产率和冻干物料的质量是冻干理论研究者和技术开发者关注的焦点^[3]。这些问题与冻干工艺的控制是否得当有直接关系。本文以某药械厂的冻干机械为研究对象,以某动物疫苗为冻干物料,分析了该冻干机的控制系统组成和控制工艺。选取对冻干能耗和冻干质量响较大的3个因素(温度、真空度、时间)进行实验研究,分析各因素对物料冻干质量的影响,并在此基础上利用三因素二次正交分析法和回归模型进行了参数优化分析。

2 冻干机控制系统构成

冻干物料控制中有两个重要的参数^[4],第一是升华结束

点的确定。升华结束点即主要干燥阶段,在冻干过程中是一个重要的转折点^[5]。因为当最后残余冰核升华后,再加入到产品的外部能量只能增加它的温度而非作为升华热,干产品的温度增高将引起崩解、产品变褐、生理活性降低^[6];相反如果产品中仍存在残余冰核时,过度增加热量将发生共晶熔化现象导致冻干失败。第二是残余水分的估计。对于大多数产品,残余水分常常在1.0%~3.0%,且随时间推移制品的活性、免疫能力、稳定性不会受到损害^[7]。但是,对于生物制品(比如本文中的动物疫苗)等的冻干对于生产质量控制提出了特殊的挑战性要求。首先,冻干状态中生物制品的稳定性与传统药物相比对残余水分的小量差别是敏感的^[8],例如冻干的生长激素在40℃后,3.0%的水分比0.5%的水分其聚合度约大5倍^[9]。其次,与低分子量药物相比生物制品对于过度干燥更易受到不利影响^[10],例如与具有较低的残余水分水平相比,残余水分为6.0%~8.0%的冻干水痘病毒疫苗产品稳定性得到改善^[11]。所以对于生物制品而言越干并不妥当。残余水分决定冻干产品的质量和加工过程的经济性^[12]。因此,冻干机控制系统主要就是控制和确定升华结束点,以及对残余水分的检测。

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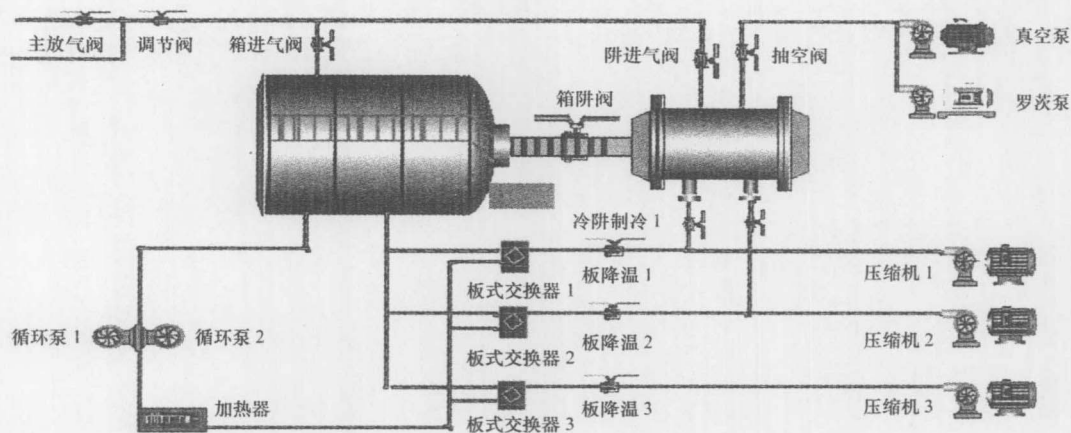


图1 冻干机原理图

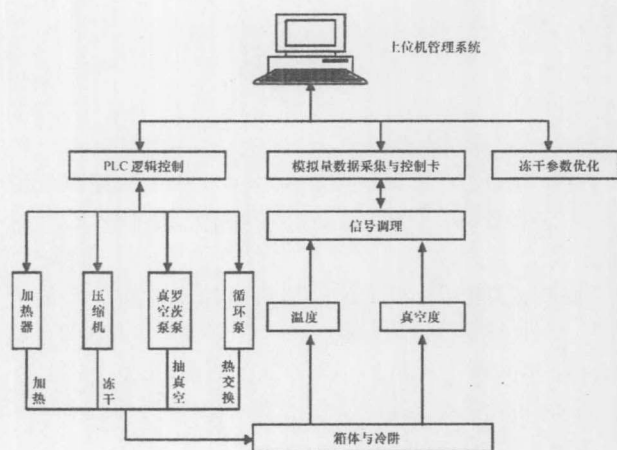


图2 冻干机控制系统结构图

在试验中,我们采用了如图1的冻干机原理图。该冻干机由干燥箱体、冷阱、压缩机、真空泵、加热器、热交换器及其他辅助设备组成。图2为冻干机的控制系统结构图,上位机负责处理和存储数据采集卡采集的现场数据,并根据采集的数据判断升华结束点和检测残余水分,并将处理结果通过一定的逻辑发送给PLC驱动各个设备(阀或泵)开启或关闭。同时,也可以通过现场PID仪表或采集卡的输出控制,控制温度和真空度。另外,上位机根据存储的数据通过一定的算法进行参数的优化。其大致工艺如下。

(1)加物料。在物料和温度探头放入干燥箱体后,关上干燥箱门,并给出关闭的确认信号给控制系统。

(2)启动制冷系统。启动冷却水。启动硅油循环泵,当一台启动一段设定时间后,硅油压力没有达到设定压力,给出报警信号,但不停机,同时自动启动另一台循环泵,停止当前这一台;当另一台经过延时后仍然不能达到设定压力,系统则给出报警信号,同时停机;此连锁在硅油正常循环过程中仍然有效。分时启动压缩机进行制冷,制冷时间和温度可设置。当达到该时间和温度后,对冷阱进行制冷。同时箱体进入低温保持期阶段。

(3)抽真空。冷阱达到设定温度后启动机械真空泵;分

别延时打开泵阱阀和箱阱阀;真空度到达控制系统设定值后启动罗茨泵。

(4)升华干燥期。(a)箱体低温保持期时间到设定值;(b)真空度到设定值;(c)冷阱温度到设定值,在满足以上条件后,加热器运行。对物料加热需注意以下问题:

- 加温时间和温度同时到达设定值判断为加温完成,进入升华过程;

- 在升华过程中,当加热温度出现失调,温度过高时(范围可调),立即切断热源,将压缩机投入对箱体的制冷;

- 当真空度过高时(范围可调),打开真空调节阀进行调节(何时进行调节可由用户根据不同物料要求进行设定)。

在升华干燥期内,根据控制系统采集的数据,如果箱体温度、真空度和低温保持期时间都达到了系统设定值,则可以判断为升华干燥期的结束点,完成对升华结束点的控制。

(5)冻干终止的判定,即残余水份的确定。判定原理为水蒸气从产品中释放出来干燥室内压力升高。关闭箱阱阀一定时间,如果压力升高说明制品水分太多;如果干燥箱体压力不变表明制品水分很少,即干燥结束。具体可以通过判断一个时间段 ΔT 内真空度 ΔP 的变化范围,其中该时间段 ΔT 和真空度变化范围 ΔP 可设定,并且判断的间隔时间也可设定。在 ΔT 开始计时时刻,关闭箱阱阀;如果判断成功,执行后续冻干终止动作,如不成功,打开箱阱阀,继续执行冻干工艺,延时一段时间后继续判断是否成功。判断成功后,用户进行确认,经确认后,结束冻干。

3 实验参数优化分析

对于一台特定的冻干机来说,其冻干面积是一定的,如本文中用于实验的冻干机面积为 10m^2 。因此在本文中冻干过程中,根据经验,影响冻干物料质量的最重要的参数就是冻干的温度、真空度和冻干时间。为此本文采用三因素二次正交回归组合分析方法^[13,14]来分析温度、真空度和冻干时间对冻干物料质量的影响。实验参数水平和参数值如表1

所示。设计冻干物料质量 5 个等级,分别为优秀(5),良好(4),中等(3),及格(2)与不合格(1)。

表 1 实验参数水平和参数值

Table 1 Experimental parameters level and value.

参数水平	温度(°C)	真空度(Pa)	时间(H)
高	-50	0	25
中	-60	1	30
低	-70	2	35

实验结果如表 2 所示。实验中有 3 个因素和 3 种水平,共需要做 $3^3 = 27$ 个实验。而用正交表只需做 9 次试验。实验结果如表 2 所示,从结果可以看出,只要冻干时间、温度和真空度在允许范围内,疫苗冻干质量基本能达到合格,但如何得到质量好的冻干物料,则必须经过实验分析。将上述实验数据保存于 EXCEL,并输入到 SPSS 软件^[15]的多元回归分析模型^[16],得到以下冻干质量与冻干时间、温度和真空度的回归模型:

$$Y = 16.4521 + 0.3024X_1 - 3.7079X_2 - 0.1424X_3 - 0.0028X_1 * X_1 - 1.397X_2 * X_2 - 0.0206X_3 * X_3 + 0.0209X_1 * X_2 - 0.0212X_1 * X_3 + 0.248X_2 * X_3$$

其中, X_1 代表冻干温度, X_2 代表真空度, X_3 代表冻干时间,取值范围分别为:

$$-70 < X_1 < -50 \quad 0 < X_2 < 2 \quad 25 < X_3 < 35$$

$F = 2.0321 > df(9, 5) = 0.2250$, 回归模型显著。F 检验说明上述回归模型的置信度在 95% 以上,能较好地反映冻干生产率。

表 2 实验结果

Table 2 Experimental data.

实验序号	温度(°C)	真空度(Pa)	时间(h)	物料质量
1	-50	2	25	2
2	-50	0	30	3
3	-50	1	35	4
4	-60	1	25	4
5	-60	2	30	3
6	-60	2	35	5
7	-70	0	25	4
8	-70	1	30	5
9	-70	2	35	3

为了获得最优化实验参数,利用前面得到的冻干质量回归模型,寻找其极值或极值点。由运筹学优化原理知,函数 Y 在区域 R 上有二阶连续偏导数,若 $\Delta Y(X') = 0$,且对任何的非零向量 $X \in R$,有 $Y > 0$,则 $X(X_1, X_2, X_3)$ 有严格局部极值。根据计算可得,当冻干温度(X_1) = -70.0000,真空度(X_2) = 1.2410 和时间(X_3) = 34.8934 时,冻干质量为 4.9864,基本达到最优。

得到最优冻干参数后,使用相同的疫苗和同样的冻干设备,经过三次实验表明最优冻干参数(冻干温度 = -70,真空度 = 1.24 和时间 = 35)可行。

4 结论

(1) 针对某 10 m² 的冻干机械,设计与开发了包括上位机、PLC、模拟量 I/O 等的控制系统,所有冻干参数通过控制

系统后,在整个冻干过程可自动完成,包括最后的冻干结束判断过程。

(2) 根据对某种动物疫苗的实验结果,建立了关于冻干质量与冻干温度、真空度、时间的二次回归数学模型,其可信度在 95% 以上。

(3) 根据上述二次回归数学模型,得到了对该动物疫苗进行冻干的最优参数,冻干温度 = -70°C,真空度 = 1.24 Pa 和时间 = 35 h)。

(4) 上述最优冻干参数只是在该 10 m² 的冻干机上针对该实验疫苗的最优参数,对于不同的冻干物料,由于其生理特性不同,必须采用本文第三节提到的方法进行实验,根据实验结果来获得合适的最优参数。

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